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Report No. IITRI-C6030-10
(Final Report)

BACKSTREAMING FROM OIL DIFFUSION PUMPS

December 1, 1963, through May 30, 1966

Task Order No. NASr 65(08)
IITRI Project C6030

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FOREWORD

This is Report IITRI-C6030-10 (Final Report) on IITRI
Project C6030, Task Order No. NASr65(08), entitled "Back-
streaming from Oil Diffusion Pumps", and covers the period
from December 1, 1963, through May 30, 1966. This work is
a continuation of "Theoretical and Experimental Study of
Various Diffusion Pump Oils" which was undertaken for Arnold
Engineering Development Center and is reported in AEDC-TDR-
63-174. The scope of the original study was increased to
include parameters of equipment, and operational procedures,
with added emphasis on the mechanisms involved. The scope
was also increased in the last period to include an eval-
uation of backstreaming contamination in turbomolecular
pumps.

The experimental work was under the direction of
W. M. Langdon and E. G. Fochtman, in the Chemical Engineering
Section, Applied Chemistry Division, of IIT Research Insti-
tute. The project was monitored by John P. Mugler, Jr.,
Langley Center, Mason T. Charak, and Conrad Mook both of
NASA Headquarters, Washington, D. C. We are indebted to the
Welch Scientific Company for the loan of the turbomolecular
pumps used in this investigation.

Experimental work was conducted by R. H. Snow,
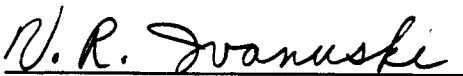D. R. Howard, V. R. Ivanuski, J. Frerichs, and H. J. O'Neill.
Data are recorded in IITRI Logbooks C14863, C14864, C14877,
and C15234.

Respectfully submitted,

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ABSTRACT

BACKSTREAMING FROM OIL DIFFUSION PUMPS

The oil diffusion pumps studied covered the variables: pumps – fractionating and nonfractionating; baffles – right angle elbow, CVC BC-61, NRC HN-6, G/P Cryosorb; oils – DC 705, Convalex 10, OS 124; and operational procedures. Optimum operating conditions resulted in no detectable increase in the 1 monolayer of oil which forms during the bakeout period. This result can be obtained routinely with either pump, all three oils and two out of four baffles.

The backstreaming contamination in turbomolecular pumps was also found to be on the order of 1 monolayer. However, the analytical methods showed the presence of an interferring substance, presumably unsaturated hydrocarbons from the lubrication process, which could not be removed by prolonged bakeout at 212°C. Backstreaming of the volatile lubrication oil can occur on startup and shutdown unless special procedures were used which involved fore pressures of several torr when the pumping rotor was below operating speed.

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PART A.   BACKSTREAMING FROM OIL DIFFUSION PUMPS

I.   INTRODUCTION

The objective of this program was to determine the amounts
and the causes of oil contamination in ultrahigh vacuum systems
employing oil diffusion pumps with baffles and stable, single-
component oils. One of the primary objectives was to determine
the operating procedures that result in minimum levels of con-
tamination.

Large space-simulation chambers can operate for long periods
without release to the atmosphere. The contamination level in
such chambers is often a controlling parameter. Vacuum systems
usually operate under less than ideal conditions with much greater
contamination levels than were measured in this program. Although
the test objects often contribute a major fraction of the dirt
load, under most circumstances a better knowledge of the cause,
cure, and prevention of backstreaming oil contamination from the
pumping system would be of great assistance in increasing the
value of the test results.

The term backstreaming usually refers to the amount of oil
that streams back from the top diffusion-pump jet and passes
through the top flange of the pump. In most installations this
massive backstreaming can be reduced 100- or even 1000-fold by
using simple traps or baffles. Although trace contamination in
baffled systems has received considerable attention, the diffi-
culty of measuring such small quantities has handicapped defin-
itive measurements. In this investigation, under optimum
conditions less than one monolayer contamination occurs after
the bakeout period in runs of over 500 hours with most combina-
tions of pump, oil, and baffle. These values are on the order
of 1/100 of recently reported measurements.

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II. TECHNICAL DISCUSSION

A. Backstreaming

Basically, backstreaming is anything that travels through a jet in a direction opposite to that of the oil vapors. Backstreaming matter can be noncondensible gases, pump oil, or degradation products, and either gaseous or liquid. In this program we were concerned with condensible material that could be collected on a cold plate in the vacuum chamber. Unavoidably, this includes adsorbed material that is collected and measured by the analytical method. The actual backstreaming process can be extremely periodic and occur as rays of matter in a completely nonhomogeneous manner. Contamination levels in monolayer equivalents are shown in Table 1 for the two oils used in this investigation.

1. Massive Backstreaming

The amount of backstreaming that occurs in unbaffled systems can be 10^6 times that in a baffled system. This large backstreaming, usually termed massive backstreaming, is defined by an AVS standard (ref. 2) as the matter that is collected at the flange entrance above the pump. Massive backstreaming is almost completely concerned with the reverse flow of hot vapors of pump oil since degradation products, both condensible and non-condensible, are present to a relatively negligible extent. Such backstreaming has a value on the order of 10^{-2} mg/cm^2-min. Many studies have been concerned with characterizing massive backstreaming, which varies manyfold, depending upon pump design, operating conditions, and oil types. (Representative values are shown in Table 2.)

Probably the most important advance in minimizing massive backstreaming is the use of a cap over the top jet. The cap is cooled either by circulating water or by conduction and radiation, and the massive backstreaming value is lowered to 10^{-4} mg/cm^2-min. This cap, which reduces the pumping speed to a negligible extent,

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is important in unbaffled systems and in those which use a
liquid nitrogen trap immediately above the pump. In the absence
of a cap the amount of backstreaming is sufficient to permit the
pump to run dry if it is continuously operated for several
months,* since the oil freezes out on the baffle.

2. Baffled Backstreaming

Baffled backstreaming can be considered as any material
that flows into the vacuum system from the pump when there is
some type of baffle inserted between the flange mouth and the
system. The baffle can be merely a length of straight pipe;
however, it is usually some sort of surface, or surfaces, on
which the backstreaming material can impinge at least once. The
capacity and the efficiency of the baffling surface are often
increased by cooling and/or adsorptive practices. A calculation
of various trap geometry versus conductances has been made by
Hablanian (ref. 20).

There is little apparent agreement in the reported values
of baffled backstreaming, probably because of the difficulty
of making definitive measurements of such small quantities of
oil. Reported measurements on baffled systems vary from
19×10^{-6} mg/cm^2-min to "no detectable deposits." The values
that are usually obtained are between 1 and 0.1×10^{-6} mg/cm^2-min.
For comparison, a recent definitive measurement by Holland
(ref. 23) reports less than 0.5 A/hr, which corresponds to
0.08×10^{-6} mg/cm^2-min.

On the basis of this program, a major source of this back-
streaming is oil migration effects; that is, there is a warm
path through a baffle along which oil molecules condense and
reevaporate. The other aspect of oil migration, i.e., travel
along the surface by wetting phenomena, is probably minor. The
backstreaming rate of unconditioned or contaminated oil is
usually quite large. In normal use, most systems tend to

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*Time for 2 cm height of liquid to backstream at a rate of
10^{-2} mg/cm^2-min = $2/(10-2)$ (10^{-3}) $(60)(24)$ = 140 days.

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condition themselves, resulting in a continual decrease in volatile contaminants and in the backstreaming rate. Hablanian's data (ref. 20) show that backstreaming values decrease slowly with long runs to values of 0.7×10^{-6} mg/cm^2-min, although the analysis is complicated by start-up effects. Langdon and Fochtman (ref. 29) obtained consistent values of 0.02×10^{-6} mg/cm^2-min over a period of months, followed by a tenfold decrease to 0.003×10^{-6} mg/cm^2-min in a long run.

3. Fore-Line Contamination

Forepump contamination can be a serious source of contamination in an ultrahigh vacuum system. Forepump oil is fundamentally a rather volatile material but can contain heavy- and light-end degradation products due to the cracking process that accompanies the various friction phenomena in the mechanical pump. The cavitation that accompanies the pumping action ejects fine droplets of oil, both upstream and downstream of the pump. Contamination is often eliminated by an intermediate diffusion pump in the fore line of the main diffusion pump. More infrequently and less reliably, contamination is minimized by cold and/or absorption traps in the fore line. In this investigation, contamination was prevented by operating the diffusion-pump fore line with a slight leak and by having the ballast on the mechanical pump slightly open. The fore line also had at least two right-angle bends.

B. System Conditions

A large number of conditions can influence the backstreaming performance of oil diffusion pumps. The complexity is too great for all of these factors to be studied in detail. By confining our attention to systems operating below 10^{-8} torr, the parameters were limited to those discussed below. The detailed process and equipment specifications are given in Section III.

1. Modern Oils

This work was restricted to two major types of stable, single-component oils: $1,1,3,4,4-$ (phenyl)$_5$-$1,3,5-$(methyl)$_3$ -trisiloxane, and mixed pentaphenyl ethers. The proprietary materials actually used were DC 705, Convalex 10, and OS 124. DC 704 [$1,1,5,5-$(phenyl)$_4$-trisiloxane] is probably the diffusion pump oil in greatest use today. This oil was tested in the early stages of our work (ref. 28) as a frame of reference, but its backstreaming characteristics were so poor compared with the other oils that it was eliminated from the program. DC 704 has been used with liquid nitrogen baffles in some of the recent ultrahigh vacuum systems because its lower boiling point is believed to cause less oil degradation.

2. Pumps

The pump, NRC HS6-1500, was specified by the original sponsor, Arnold Engineering Development Center, because it was the only available United States pump with a cap over the top jet at that time. This cap was an essential requirement for Arnold's con- templated long-term operation with a liquid nitrogen baffle directly above the pump. In the absence of a cap, runs would be limited to less than 100 days because of the oil freezing out on the baffle. A baffle arrangement without an intermediate water-cooled baffle would give maximum conductivity and pumping speed at the expense of greater liquid nitrogen consumption. This pump employs a two-compartment boiler and can be considered representative of a fractionating pump.

The CVC PMC-1440A pump, which also has a cap over the top jet, was chosen as representative of a completely different type of pump that receives widespread use in the United States. This pump employs multiple, siphon-type boilers in the vaporizer, which is not compartmented. This pump is nonfractionating in the conventional sense, however, fractionation can be obtained by adjusting the exit cooling water close to the boiling point. Consequently, the hot condensed oil entering the boiler contains

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a minimum of dissolved gases and volatile components. A major difference between these two pumps is that the PMC-1440A does not have an ejector jet; however, it does contain four diffusion jets, whereas the HS6-1500 pump has three.

3. Baffles

The original selection of baffles involving a one-bounce, right-angle elbow and the CVC-BC-61 chevron trap was somewhat arbitrary. The main purpose of the program was to evaluate the backstreaming characteristics of the two recently available pump oils, and these baffles were selected for test as representative of one- and two-bounce types.[*] The two other baffles, NRC-HN-6 and G/P-Cryosorb-251, were later included as representative of other major industrial baffles.

Baffle temperatures of -75°F were used in the majority of the tests since they could be reliably and economically maintained for months by mechanical refrigeration without any attention. Spot tests at temperatures of -25°F and liquid nitrogen indicated that the same results were obtained as at -75°F. The BC-61 and the HN-6 baffles which have warm walls and might allow oil migration, were tested while the sides were cooled with external cooling coils.

4. Seals

Double elastomeric 0-ring seals with guard vacuum were employed throughout the program. It was determined early that unlubricated silicone seals, after a short run-in period, would not contribute any detectable contamination. This sealing technique was extremely helpful in simplifying our measurements, which involved numerous and rapid assembly and dissembly of the

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[*] These terms do not define the trapping characteristics as well as transmission probability and average number of contacts (see Table 6).

system to remove the sample from the collection plate.

5. Operating Conditions

A conditioning period of at least 2 weeks and sometimes 1 month was required before definitive results could be obtained. Once obtained, a system generally remained conditioned (i.e., gave low values of backstreaming) or recovered rapidly from mild contaminant loads. If a system did not start to perform satisfactorily in 2 weeks, the best solution was a complete cleanout and oil change.

A detailed set of standard operating procedures was developed during the course of this work. These procedures were found necessary to obtain reproducible low values and were specific for this purpose. These procedures are discussed in Section IIIB.

C. Theoretical Considerations

The fundamental mechanisms that cause the trace contamination measured here are unknown. For example, all of the measurements in AEDC work (ref. 28), except the last (ref. 29), were rate dependent and very reproducible. It is now known that the higher values for those stations employing BC-61 baffles were due to oil migration along the warm sides of the baffle. The values for the stations with the elbow baffle must be due to some other source since oil migration effects along warm sides are absent. Explanations can be postulated which suit most of the available evidence, however, further verification is needed.

In a system with a correctly designed baffle, all vapors will contact a cold surface at least once. The backstreaming value will then depend upon the sticking coefficient. A baffle that initially allows considerable oil to backstream will, after conditioning, perform with a coefficient as close to unity as it is possible to measure. The sticking coefficient, or the contacting phenomena, has obviously undergone a radical change. It is probable that backstreaming is caused by periodic, non-homogeneous phenomena, from which mechanisms can be postulated

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which explain this behovior.

The fundamental causes of backstreaming in well-baffled systems are undoubtedly related to the violently explosive characteristics of the vaporization processes that occur in the diffusion pump. The most obvious is the eruptive boiling process that takes place in the main boiler. Related to this is the explosive revaporization of any light-end oil droplets that contact the hot external surfaces of the jet stack or the oil in the bottom annular space. Leaks or gas evolution will usually take place as a series of explosive pulses. Thus such violent processes can give rise to infrequent bursts of vapors. It is very improbable that any measurement in the gas phase would be statistically significant as an indication of back-streaming. If the backstreaming material consists of a rela-tively large liquid drop, one such particle will constitute appreciable contamination over a period of days. Such contam-ination may not always be detected by pressure or mass spectro-meter measurements. Conditions are ideal for such liquid drops to exist for days in a superheated state without exerting appreciable vapor pressure.

It has not been demonstrated whether gas or liquid, or both, are involved in baffled backstreaming. We are inclined towards the viewpoint that liquid droplets are often involved. This work showed that a system with a simple elbow baffle will backstream less than 1 monolayer in run periods up to 544 hr. The same value was obtained for the widely different baffle temperatures of -25 and -75°F. This demonstrates that the accommodation coefficient for the oil vapors is as close to the ideal value of unity as possible. In systems that gave up to 50 monolayers in 70 hr, trap temperatures down to liquid nitrogen had no measurable effect on the amount of backstreaming. Ricocheting of small liquid drops could be responsible for this relatively great amount of contaminant penetration. This ricocheting has been observed in high-speed

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cinephotomicrography of a similar process, the coalescence of very small liquid drops on liquid surfaces (ref. 27).

Backstreaming could be entirely due to gaseous backstreaming. In an analysis (ref. 25) of the contribution of intercollisions in the gaseous state to backstreaming, the calculation for 10^{-8} torr gave the very low value of 10^{-10} mg/cm^2-min for both oil-oil and oil-gas collisions in an elbow trap. These very small values are obviously unrelated to the value of $>10^{-7}$ mg/cm^2-min usually obtained. However, if we have an infrequent ray of gaseous material with localized pressures approaching 1 torr or higher, such a ray in a one-bounce trap could give rise to gaseous intercollisions and account for the backstreaming, since some fraction of the gaseous ray will not contact the baffle surface. It is of interest to note that a sticking coefficient of 0.9 corresponds to a backstreaming rate of 10^{-7} g/cm^2-min through an elbow baffle by the calculations of Jones and Tsonis (ref. 25).

The viscosity of the oil can be related to much of the observed backstreaming phenomena (ref. 28). In the previous work one of the most striking differences was that under similar conditions the right-angle elbow was usually ten times more efficient than the chevron baffle. Another difference was that pentaphenyl ether was usually ten times better than DC 705. These differences occur when backstreaming was appreciable. In this program, under optimum conditions the same low value was obtained for both oils or both traps. An apparent explanation is that the right-angle elbow does not present an opportunity for liquid to drop onto a hot surface and undergo explosive re-vaporization. Under similar conditions the more viscous penta-phenyl ether should have less tendency to drip. The explosive revaporization process requires a more volatile material in the drop than in the bulk oil. This situation can occur if the drop absorbs sufficient gas before dropping off the surface. Visual

observation shows some of the drops persist for hours or days before they leave the baffle surface.

D. Measurement of Oil Deposits

It is rather difficult to fully define many of the reported values of backstreaming in baffled systems and to compare them with this work. Analytical techniques have been inadequate, and there has been periodic mention of "no contamination" in systems that probably had very high contamination levels. Fedor and Koontz (ref. 15) described a wettability technique and gave data to demonstrate sensitivities of 0.1, 0.5, and 3 monolayers for fatty acids, lard oil, and mineral oil, respectively. Although this method could be extremely useful and generally applicable, it was found to be nonreproducible in our work (ref. 28), and sensitivities generally were greater than 25 monolayers with DC 705.

Holland (ref. 23) described a method that consists of polymerizing the deposit with an electron beam and determining the deposit thickness by reference to a masked section by using a multiple-beam interferometer. This technique gave absolute measurements of 50 A (5 monolayers). Holland reported backstreaming values of <0.5 A/hr for a DC 705 (<1 x 10^{-8} mg/cm^2-min) system trapped with liquid nitrogen.

Carter et al (ref. 5) used radioactive tracer techniques that detect deposit formation of <0.4 A/hr by measurements at 5-min intervals. This special short-term technique is only suited to very special applications.

Hablanian (ref. 20) has recently used a solvent technique for removing the deposit and weighing the change in weight of the collecting surface. He determined values of 5 x 10^{-6} mg/cm^2-min for DC 705 and 0.4 x 10^{-6} mg/cm^2-min for pentaphenyl ether. These values were continually decreasing with run time, and a final value of 0.07 x 10^{-6} mg/cm^2-min was obtained with DC 705.

Actually, many sensitive analytical techniques could be used in this application. (Some of the available techniques are listed in Table 3.) These methods are fundamentally capable of good accuracy in the ppb range. Theoretically, ultrasonic techniques can detect weight changes down to 10^{-12} g in a 1.0 g total weight. Measurement of distance changes of the order of 10^{-10} cm are also possible (No. 8 in Table 3). All of these methods would require considerable development work before useful and definitive values could be obtained. Reproducible values should be accepted as valid only after they have been obtained by different methods. The agreement of two methods that are based on independent principles is considered verification of the value. Most of these sensitive analytical techniques depend on a measurement of a physical property that is empirically correlated to the chemical structure, and very slight changes in the system can destroy the correlation.

One of the most universal and sensitive analytical tools today is gas chromatography. It was only at the end of this work that a technique was developed to analyse DC 705 deposits of 10^{-3} mg, which corresponds to 0.1 x 10^{-4} mg/cm^2 (0.1 monolayer) in our work. A concentration of 1% DC 704 can be determined quantitatively, in these small deposits. The use of tracers would increase the sensitivity to 10^{-5} mg. Even these values represent only 1/40 of the theoretical limit of instrument sensitivity. Sensitivities of 10^{-4} mg have been obtained on known hydrocarbons (Item 7 in Table 3) at IIT Research Institute.

III. EXPERIMENTAL WORK

A. Test Stations

As shown schematically in Figure 1 and as pictured in Figure 2, five test stations were set up. All diffusion-pump stations were instrumented to failsafe in case of failure of water, electricity, or vacuum. All major components were selected to be representative of commercially available equipment. The equipment was used without modification, except that the sides of some of the traps were cooled externally to eliminate oil migration along the warm walls. Data on the mechanical pumps, diffusion pumps, baffles, oils, collection plates, and refrigeration are detailed in Tables 4 through 9 and Figures 3 through 9.

The mechanical pumps were operated with the ballast valve open 3/4 turn at all times. All stations, except No. 1, were operated with an air leak at the fore-line flange of the diffusion pump sufficient to maintain the fore-line pressure at approximately 20 μ. Station 1, without a fore-line leak, was operated near 1 μ. This procedure combined with the two elbows in the fore line minimized contamination by mechanical pump oil.

The diffusion pumps were operated with rated power except for the programmed start-up procedure (Table 18). Tests with power input varying from 75 to 125% of rating showed no effect on backstreaming. The HS6-1500 pumps were operated with the cooling water valves wide open. Consequently the exit cooling water temperature varied from 35 to 65°F with winter and summer. The PMC-1440A pump was operated with the exit water thermostated to 200°F, as recommended by the manufacturer.

All main seals were designed with double silicone O-rings, which were unlubricated and utilized a guard vacuum. The O-ring groove specification (Figure 10) permitted the silicone O-rings to be used repeatedly without being disturbed when samples were removed. Other elastomeric materials such as Viton, buna,

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neoprene, and butyl, were much less satisfactory due to trace contaminants and adherence to the metal. When experimental changes resulted in the use of a seal with a single O-ring groove, the O-ring was lubricated with Apiezon H. This grease did not contribute any material that noticeably influenced the ultraviolet absorbance readings at the wavelengths of interest.

Refrigeration for the traps and the collection plates was generally -75°F but was varied from room temperature to -125°F. Cooling was provided by mechanical refrigeration (Table 9), and methanol was used as the circulating coolant.

Pressures were read with the ionization gauges, CVC-GIC-017 and Varian UHP-12P, and the gauge control Varian 971-0003 (Table 11) was used. All gauges used 1-in. tabulations and were provided with traps (H. S. Martin Company, Cat. No. M-40450, 500 ml) so that readings could be made at room temperature or with liquid nitrogen trapping. The partial pressure analyzer used for this work was the General Electric partial pressure analyzer model 22PC110 (Table 12). Permanent magnets of 2.5 and 5.0 kilogauss were used. (Complete details on the gauges and the analyzer are given in Tables 10, 11, and 12.)

The backstreaming oil was collected on a refrigerated plate located just above the baffle (Figures 8 and 9). The amount of deposit was determined by washing off the oil with a solvent and analyzing by ultraviolet absorbance and later by gas chromatography.

The various pieces of equipment were used in many different arrangements over a period of four years; the arrangements of the five stations are shown in Table 13 through 17 so that questions concerning specific runs can be resolved by reference to the appropriate station.

B. Experimental Procedures
    1. Standard Conditions
    During this program very low backstreaming values were

obtained consistently. This is a direct result of experience
with the system and conditioning of the oil. These low results
required a continual reexamination of our collection and analysis
techniques. Consequently, a standard procedure (Table 18) was
established to minimize the variation in data for such small
quantities of oil. Selection of these conditions constituted
an important part of the results of the program and are discussed
later.

2. Analytical Techniques

Quantitative determination of the amounts of oil deposits
were generally made by ultraviolet absorbance measurements,
since this method is simple, fast, and nondestructive. The
determination by gas chromatography is essentially the opposite.
Moreover, it was not until the end of the program that gas chroma-
tography was developed to a usable extent. Gas chromatography
is much more sensitive and informative as to actual components
than the ultraviolet method. The greatest use of gas chroma-
tography requires tracer techniques that were not developed here
due to lack of time.

3. Solvent Treatment of Collection Plate

The oil deposit on the collection plate was removed by
washing the plate with a solvent. Originally a selected lot of
methanol that had been checked for satisfactory ultraviolet
absorbance characteristics was used. Later, no selection of
solvent was necessary. Instead, a reference sample of solvent
was taken and used for each set of plate samples. The volume
of solvent was gradually reduced from 50 to 10 ml. In initial
experiments the collection plate was rinsed with copius amounts
of alcohol and replaced for the next run. In later work, the
plate was washed copiously three times, and then a blank sample
was obtained using 10 ml of solvent. This was followed by three
more washes and a second blank sample.

The rinsing process collected 95% of the oil under most
circumstances; this was more than adequate. Attempts were made
to collect the sample in large quantities of solvent and

concentrate it to a few ml. The amount of background that developed in the concentration step generally prevented satis-factory measurements.

4. Ultraviolet Absorbance

The ultraviolet absorbance value was determined for the samples and compared with fresh solvent by using a Cary spectro-photometer model 14. The comparison solvent was drawn fresh from the same solvent bottle each time a plate was rinsed. The ultraviolet absorbance was measured in 1-cm curvettes, which were isolated from all other uses, and separate sets were used for each kind of pump oil.

The amount of oil deposited can be calculated from

$$W = Vm \ A.U.$$

where

W = total weight of oil on the collection plate, mg

V = total volume of rinse sample, ml

m = slope of calibration curve, mg of oil/ml/A.U.; equal to 0.39 for DC 705 and 0.063 for Convalex 10 or OS 124

$A.U.$ = absorbance units measured at 258 and 278 mμ for DC 705 and pentaphenyl ether, respectively

The accuracy of the data (Table 19) was verified by seeding the plates with known weights of oil (Table 20). The analytical blanks for pentaphenyl ether were 0.5×10^{-4} mg/cm^2 or lower, whereas for DC 705 they were usually 1 to 2×10^{-4} mg/cm^2. These blanks indicated that pentaphenyl ether deposits could be measured to an accuracy of less than 1×10^{-4} mg/cm^2 whereas DC 705 could be measured with an accuracy between 1 to 2×10^{-4} mg/cm^2. (Figures 11 and 12 show the actual curves for deposits of 1×10^{-4} mg/cm^2 and higher. With small amounts, under 5×10^{-4} mg/cm^2, the absorbance spectra had no characteristic peaks. The validity of the measure-ment was a major result of this work and is discussed in Section IV.

5. Gas Chromatography with Pentaphenyl Ether.

Development of a chromatographic analysis of pentaphenyl ether was undertaken early in the program. Later, somewhat different methods were developed for silicone and turbomolecular pump oil.

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Data on used oils and backstreaming deposits are discussed in Section IV.

The methanol elutriates of pentaphenyl ether derived from plate washings were concentrated to near dryness at room temperature and dissolved in approximately 0.1 ml of benzene. Approximately 10 μl of this solution was used for analysis. The fresh oils were prepared for analysis by diluting to 10% oil in benzene and using 1 μl of the resulting solution.

All analyses were performed on a Loenco model 70 gas chromatograph employing a dual hydrogen-flame ionization detector. The column consisted of a 6 ft x 1/8 in. copper coil containing Chromosorb ABS (120 to 130 mesh) coated with 5 wt. % SE-30 silicone gum. The helium carrier gas was maintained at an inlet pressure of 65 psig to maintain an exit flow of 40 ml/min. The signal was directed to a 1-mv recorder with a full-scale range of 8 x 10^{-10} amp. The temperatures of the column, detector, and injection port were maintained at 265, 280, and 275°C, respectively.

No direct quantitative calibration was made for pentaphenyl ether since the accuracy of the much simpler ultraviolet method was more than adequate. A qualitative comparison with ultraviolet and weight is given in Table 21. The actual curves (Figure 23) are discussed later. These data give an accurate indication of the amounts of nonpentaphenyl ether constituents. As shown below for DC 705 and turbomolecular pump oil (Part B), gas chromatography can be extended to considerably greater sensitivity than ultraviolet. The latter, though much simpler, uses a dilute sample that must be concentrated for gas-chromatographic analysis. However, sample concentration for the ultraviolet method has, to date, been unsuccessful because of the large increase in background, which makes the ultraviolet reading unusable.

6. Gas Chromatography for DC 705 and DC 704

The gas-chromatographic analytical technique was developed to aid in evaluating the performance of DC 705 oil containing

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small amounts of a well-characterized light-end component (actually DC 704). The ultraviolet technique did not distinguish between these similar compounds. Moreover, the quantity of oil ($\sim$0.02 mg on a collection plate of 179 cm^2 is equivalent to 1 monolayer) was so small that the ultraviolet spectrum did not show characteristic peaks (Figure 11).

The methanol elutriates from the plate washing were concentrated in a Teflon test tube shaped to a 60° cone by passing a room-temperature stream of dry air or nitrogen over the sample until the solvent had evaporated. Ten μl of benzene were added to the residue, and the entire liquid used for analysis. The recovery of oil on known samples was 76% (Table 22), which was more than required since the oil deposits can vary by 100%. Obviously, the use of an internal standard would increase the precision. Samples of the unused oils were prepared by diluting to known volumes in benzene and injecting from 0.5 to 5 μl as desired.

Initially, a 6 ft x 3/16 in diameter copper column was employed. The packing consisted of 3% SE-30 on Anskrom ABS 120 to 130 mesh with a helium pressure of 26 psi. The final column consisted of 5% SE-30 on Anskrom ABS 60 to 70 mesh with a helium pressure of 38 to 40 psi. Temperatures of the column, detector, and injector were 300, 310, and 315°C, respectively.

Calibration curves for DC 705, DC 704, and mixtures of each are shown in Figures 13, 14, and 15, respectively. The data on DC 704 and DC 705 mixtures are summarized in Figures 16 and 17.

Data for used oil and for backstreamed samples are discussed in Section IV. The validity of the measurements for DC 705 appears to be better than 0.1 x 10^{-4} mg/cm^2.

7. Mass Spectrometry

The general equipment and conditions used in the mass spectrometer tests are listed in Table 12. These conditions give maximum definition for m/e = 28 (N_2) and were used in all tests unless stated otherwise. The mass spectrometer tube was inserted radially into the test spool through a 1-in opening. It was sealed by double silicone O-rings provided with a guard vacuum.

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The tube was baked out repeatedly for periods of 2 to 18 hr at temperatures of 250°C.

8. Calculation Methods

The sensitivity of the partial pressure analyzer for a particular gas is defined as the ratio of the response of the analyzer (in amp) to the actual pressure of the particular gas in the vacuum system. There are three ways to determine the actual partial pressure. One method is to leak the gas to be measured into the system at a rate such that it is the main gas in the vacuum system and then determine its pressure with a Bayard-Alpert gauge. When the leak rate is not sufficient to raise the system pressure 100-fold over the base pressure, the gas in the system cannot be assumed to consist entirely of the leaked gas. The pressure is corrected by subtracting the no-leak Bayard-Alpert reading from the with-leak reading. The result is then multiplied by the Bayard-Alpert gauge calibration factor for the leaked gas.

A second method is to introduce the gas as a controlled small percent of a carrier gas such as air or nitrogen. This method is an indirect comparison of the Bayard-Alpert reading with the minor constituent which is calculated from:

$$P_x = P_{carrier} \, X_x \, \sqrt{\frac{M_x}{M_{carrier}}}$$

where

P = pressure, torr

X = mole fraction

M = molecular weight

subscript x = minor gas constituent.

The square root expression gives the theoretical effect of pumping speed on the pressure in a system of gases of different molecular weights.

A third method employs a calibrated leak, V, into the system.

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$$P_x = 500 \sqrt{\frac{M_x}{28}} \left[\frac{V}{(60) \ (1000)} \right]$$

where

V = leak rate, ml/min

500 = pump speed for nitrogen, liters/sec

28 = molecular weight of nitrogen.

By measuring the pressure with the Bayard-Alpert gauge, this equation can also be used to calibrate the leak. Sensitivities for the mass spectrometer are summarized in Table 23 and complete data are listed in Section IV G and Table 50.

9. Pressure Measurements

The work prior to October 1964 employed ionization gauge tubing 23 mm I.D. x 3-in long, which did not have provision for liquid nitrogen trapping. Since October 1964 all pertinent readings were made with trappable gauges which employed an H. S. Martin spherical trap (500 ml sphere with internal spherical reservoir, 1/2 in clearance, 3-in tubulation on each end). (These traps are pictured in Figures 2 and 3.) The pressure readings are differentiated in the tables by RT for room temperature and by LN_2 for nitrogen cooling. This system was very informative in this program. The light ends*present due to operation with cooled baffles often result in room temperature readings of 10^{-7} torr versus liquid nitrogen readings of 10^{-9} torr. Stable and reproducible readings cannot always be obtained on DC 705 systems without liquid nitrogen trapping.

No gauge calibrations were made since we are concerned only with pressure ranges. By employing their nominal or calibration emission current, all gauges were found to agree within 100%. It was previously determined (ref. 28) by routine checks against a relatively unused standard gauge on the same tubing that even a severely discolored gauge tube did not change its calibration in the worst case by more than a factor of two.

It was observed that untrapped gauge readings on DC 705

*Mostly water.

systems were inconsistent. All gauge readings were made with at least 1/4 hr degassing by ion bombardment (Varian control) and a waiting period of at least 1 hr. Usually on alternate days a room-temperature gauge was read in the morning after an 18-hr waiting period. Variations of consecutive readings became pronounced only with DC 705. Investigation* revealed that such untrapped gauges, after degassing and a 1-hr wait, would read one decade lower than a degassed liquid nitrogen-trapped gauge. The untrapped gauge reading would then start to rise and in a period, which varied in an uncharacterized manner from 1 to several hr, balance out one decade above the liquid nitrogen-trapped gauge reading.

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*Station 2, operating with room-temperature baffles and using individually tubulated gauges. Presumably very small quantities of light-end oil molecules were present.

IV. RESULTS

A. Backstreaming Contamination under Standard Conditions

As shown in Tables 24 through 28, under standard and optimum conditions backstreaming was less than one monolayer, 1×10^{-4} mg/cm^2, after bakeout during runs of over 500 hr. This performance was obtained with either pump and either oil and two out of four baffles. The data (runs 139, 151, 137, 142, and 143) show that the monolayer present at zero time is adsorbed during the bakeout period and is not removed by temperatures of 212°F for Convalex 10 or 470°F in the case of DC 705 but is removed by the solvent wash. The analytical measurement leaves no doubt that this basic contamination level is a definitive amount. These results show that a clean and conditioned oil-diffusion-pump system can be operated quite routinely for extended periods without a detectable buildup in vacuum chamber contamination level.

B. Validity of Measurements

The entire value of this work depends upon the validity of the analytical measurements. Much of the difficulty in the past has been due to the analytical procedures employed. It was necessary to expend an appreciable part of the effort on the analytical technique, and continual refinements were developed throughout the program. It is believed that the importance of the subject merits further discussion.

1. Ultraviolet Absorbance

In Section IIIB calibration data on absorbance versus weight for the different oils were presented. As shown by the data (Table 19), agreement was within 1%. Simulated measurements obtained by seeding collection plates with known amounts are shown in Table 16. The major deviation that occurred with the lower amounts of oil (1 monolayer, 1×10^{-4} mg/cm^2) is to be expected. These values were all obtained by reference to an

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analytical blank. Note that this is an automatic overall check on the analytical method and indicates any errors in procedure, sampling, collection, instrumentation, etc., that do not remain constant and that constitute background noise. The DC 705 oil shows a much greater and more variable blank than the pentaphenyl ether. The measured values for the seeded DC 705 samples and the analytical blanks generally showed a sharp and pronounced jump of 3 ± 1 monolayers over the seeded amount. This phenomenon cannot be explained since new or well-rinsed plates should show a negligible blank. The differences were, however, very reproducible.

The analytical method for pentaphenyl ether was checked by gas chromatography, ultraviolet absorbance methods, and weight in several runs to determine the presence of light ends. The data (Table 21) indicated very close agreement between the three methods except for run 57. In this run, the weight analyses did not indicate as many light ends as gas chromatography.

The gas-chromatographic technique was not developed further for pentaphenyl ether. The ultraviolet absorbance method, as exemplified by the behavior of the analytical blanks, showed that the results for pentaphenyl ether were consistent within an average deviation of 0.2×10^{-4} mg/cm^2.

The ultraviolet spectrum for 1 to 4 monolayers did not show any distinctive peaks (Figures 11 and 12). In addition, the ultraviolet spectrum did not distinguish between similar compounds.

2. Gas Chromatography

Gas-chromatographic techniques were developed for mixtures of DC 705 and DC 704. The data (Tables 19 and 20) indicated a sensitivity of at least 10 times and probably 100 times that of the ultraviolet method. Moreover, chromatographs identified distinctive peaks for the DC 705 and DC 704, whereas indefinite traces were obtained by ultraviolet methods. As shown in Table 35, run 232, values of 2.40×10^{-4} and 2.1×10^{-4} mg/cm^2 were

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obtained by ultraviolet and gas chromatography, respectively, and the amount of DC 705 present was 89.5, 89.4, and 93.3% for runs 232,234, and 239, respectively. There is no doubt that these deposit values, on the order of 1 monolayer, are a true and valid measurement of the contamination; however, there are some unidentified light-end components present (Peak A Figure 24).

C. Pump Performance

The two types of pumps tested were the NRC-HS6-1500 and the CVC-PMC-1440A (Table 5), which are representative of two common types of industrial pumps. The results (Table 24 through 28) indicate that under optimum conditions both pumps gave the same low backstreaming value of $\sim 1 \times 10^{-4}$ mg/cm^2.

Considerable time and effort were required to get the PMC pump on the line; this was probably a contamination problem since a similar phenomenon occurred with two out of four stations that employed HS6-1500 pumps. The PMC-1440A pump was operated with 200°F exit water and a fore pressure of 10^{-3} torr because this condition prevailed when optimum results were finally obtained. The HS6-1500 pump was operated satisfactorily without any particular control of the cooling water or the fore pressure. Consequently, the exit cooling water for this pump varied from 35° to 65°F from winter to summer. The fore pressures were usually 1 to 2 $\times 10^{-2}$ torr and infrequently 6 $\times 10^{-2}$ torr.

The high temperature of the exit water for the PMC-1440A pump is recommended by the manufacturer to decrease the gas solubility of the oil returning to the boiler. This high temperature serves the same purpose as the fractionating process in the compartmented boiler of the HS6-1500 pump. From the standpoint of removing light-end components, which result from either the load in the test space or from oil decomposition, operating procedures involving fractionation should always be employed. Light ends are usually accompanied by increased backstreaming contamination.

This work did not indicate any difference in the operating procedures for the two pumps per se; however, these procedures were not investigated specifically because of emphasis on other factors. When operating a pump for minimum contamination, there is little reason to tolerate exceptional operating conditions. Undoubtedly, under exceptional conditions differences will occur. For example, the HS6-1500 pump with 4×10^{-1} torr of air in the fore line and zero through put gave a pulsing fine pressure and a 13-fold increase in oil deposit. In this case the pumping action was apparently unstable, even though the fore-pressure tolerance for DC 704 is given as 0.7 torr. Presumably, a different heat input and cooling water temperature would allow the same fore-pressure tolerance with this oil. This type of backstreaming contamination might be related to variation in the start-up backstreaming deposit with heat input. The data show a minimum deposit with 50% heat input on the HS6-1500 pump. This phenomenon was not tested on the PMC-1440A pump, although its thermal siphon heaters could be expected to have different behavior than the more usual flat-bottom surface heaters that are used on the HS6-1500 pump.

The PMC-1440A pump did show the phenomenon of a system pressure (RT) of 10^{-8} torr that had slow pressure excursions into the 10^{-7} torr every 3 to 4 min. This pressure excursion was not noted with a liquid nitrogen reading near 10^{-9} torr. It was thought this was due to the variable evolution of light ends due to the type of fractionation employed in this pump. However, the same P_{RT} excursion was found in the final tests on Station 4, which used the BC-61 trap from Station 1. It is therefore possible that a methanol leak in the trap was responsible for the excursions and that the same leak initially larger but partially plugged in use may have been responsible for the difficulty in getting Station 1 on the line. However, careful helium leak tests on Station 1 disclosed no signs of a leak.

D. Pump Oils

1. Optimum Conditions

Three oils were evaluated during the program: DC 705, Convalex 10, and OS 124. Under optimum conditions the amount of backstreaming from these oils was identical. Thus, with oils used in a well-trapped system and with good operational practices, the backstreaming deposit was about 1.0×10^{-4} mg/cm^2 which is equivalent to one monolayer of oil. (Typical results are given in Table 24 through 28.) The results for DC 705 showed slightly more variability than the results for pentaphenyl ether because of the greater variability in the analytical blank, which in turn is related to the greater tendency of this material to adsorb on surfaces.

2. Stability under Normal Operating Conditions

Visual examination of oils after 2 years of continuous operation without any abuse show that DC 705 was unchanged but pentaphenyl ether was slightly yellow, and there were small dark flakes floating in the bulk liquid. Examination of the pump stack (Figures 18 through 20) showed that only pentaphenyl ether caused dark deposits on the external surfaces of the stack. These deposits were undoubtedly due to oxidative degradation. DC 705 could have undergone the same phenomenon, but its decomposition products were usually colorless. In the worse case for pentaphenyl ether the amount of degradation was of negligible influence and should cause no appreciable difficulty for many more years.

Catalytic decomposition of the oil apparently occurred in Stations 3 and 4 (Table 29 and 30). The tests were primarily concerned with evaluating the HN-6 baffle and several of the factors involved were not appreciated until later. The data indicated that the defective baffle (HN-6D, run 133) gave the same performance (7×10^{-4} mg/cm^2) as obtained with a new HN-6 baffle (runs 223 and 238). Apparently, the presence of methanol and nickel particles that sloughed off of the plated trap had

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no effect. There were undoubtedly hydrogen bursts due to decomposition (methanol or nickel-catalyzed oil decomposition) prior to run 133, but they were extremely infrequent at this point. However, the phenomenon became more pronounced between runs 164 to 186 and, as shown by runs 205 and 210, may have been due to the corroded jet stack. It appeared likely that the cleaning cycle, which involved refluxing the system with methanol caused the excessive corrosion of the aluminum stack and that the corroded stack caused catalytic degradation of the oil.

It is believed that some type of catalytic degradation was responsible for the backstreaming values obtained in Station 4 with the right-angle elbow (runs 34 to 134). The pressure readings showed the same sharp periodic pulses as in the case of Station 3 for runs previous to run 133. A cleaned system with new oil gave a value of 3×10^{-4} mg/cm^2 (runs 237, 240, and 244) which was very close to the optimum value of 1×10^{-4} mg/cm^2.

3. Effect of Air Leakage on Stability of Pentaphenyl Ether.

An exploratory study of the effects of air leakage was made with the mass spectrometer. It was not expected that quantitative results would be forthcoming, since the stability of the oils appeared to preclude this. Rather, it was hoped that patterns of the mass spectra and their changes with various leakage conditions could be used to monitor these effects. Their rate of formation and the disappearance of the various masses should constitute an indication of their importance.

Mass spectra for Station 5 are shown in Figure 21. Blanks in the mass numbers do not necessarily indicate missing peaks since they may or may not have been measured. There is usually considerable background noise in the lower 10^{-12} amp region. Consequently, only mass peaks that have other lower peaks on both sides can be used as evidence of a true mass peak in this amp region.

Prior to these particular tests the station had been subjected to various regimes that cannot be well defined. Station 5 was subjected to undefined air leakage for more than 2 months.

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The system pressure varied between 10^{-7} and 10^{-8} torr, which serves as a rough guide to the extent of the leakage. The mass spectra shown involve periods of 3 days, during which ambient air was leaked into the test heads of the station. The magnitude of any leak can be readily determined from any system pressure by using the net pumping speed of 500 liters/sec at the pump inlet for nitrogen.

The room-temperature baffle with an air leak (Figure 21A) shows numerous masses over 60 in the 10^{-11} amp region and one very predominate peak at 76 (or 78) of 10^{-9} amp. With the baffle at -75°F all peaks over 50, including 76, decrease to the low 10^{-12} amp region (Figures 21F and G). With air off and with the baffle at either room temperature (Figure 21C) or -75°F (Figure D & E), all heavy mass peaks decrease essentially to zero.

The rapid decrease of these peaks with the leak off indicates that they are due to air degradation and that such degradation is minor and self-purging.

Since the vapor pressure of benzene at -75°F is 10^{-4} torr, the effect of baffle temperature in decreasing the mass peaks indicates that these are degradation products of fairly high molecular weight.

4. Eruptive Boiling and Revaporization

These phenomena are sometimes considered a major contributing factor to backstreaming. This is obviously true in massive backstreaming. No direct experiments were performed on these factors; however, some observations may be of interest. In small glass or metal pumps, the apparent eruptive boiling process is quite violent for both oils; however, pentaphenyl ether appears to be much more violent. The apparent increase in ebulation violence for pentaphenyl ether relative to silicone oil is a phenomenon generally true for more viscous materials and has been recognized in vacuum distillation techniques for many years. The eruptive boiling in any 6-in metal pump is not evidenced by noise, although there is visual surging in the level indicators connected to the

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pump vaporizers.

Conditions for eruptive revaporization are unusually favorable in most diffusion pump setups. Any liquid collecting on a baffle whose temperature is at ambient or below can absorb gaseous material and, upon dripping onto a hot stack surface, revaporize explosively. The greater fluidity of DC 705 and the greater opportunity for dripping on a hot stack with a chevron baffle give results* in agreement with this postulation, namely, 10 times greater values with DC 705 than with pentaphenyl ether and also 10 times greater with the chevron than with the elbow baffle. Such revaporization may project liquid drops upward and seems a much more probable cause of liquid or gaseous backstreaming than eruptive boiling in the pump boiler. The baffle-temperature independence of backstreaming in an unconditioned system indicates that it is more likely liquid drops, since the sticking coefficients for liquid drops should be totally unrelated to that for gaseous molecules and independent of baffle temperature. As mentioned previously, concentrated rays of gaseous molecules could have an apparent sticking coefficient under unity because of the localized intercollisions that prevent some molecules from hitting the first surface. Two or more bounce traps should destroy such rays and trap the gaseous molecules. Liquid drops, however, even if occurring very infrequently, could ricochet repeatedly off successive surfaces.

5. Comparison of Convalex 10 and OS 124

Most of the results with pentaphenyl ether were obtained by using Convalex 10, a pentaphenyl ether subjected to a proprietary treatment and sold specifically as a diffusion-pump oil. Some of the earlier work (ref. 28) used OS 124, which is generally employed as a high-temperature lubricant. Runs made after replacing the OS 124 with Convalex 10 showed a general improvement in backstreaming performance. This was probably due to improvements in experimental techniques since the replacement of

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*Under non-optimum conditions when rate dependent backstreaming is taking place. **IIT RESEARCH INSTITUTE**

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Convalex 10 by OS 124 in Station 5 gave the same optimum value of 1×10^{-4} mg/cm^2 (Table 24).

The distribution of the major isomeric components as found by gas chromatography for unused OS 124 and Convalex 10 oils peaks are shown in Figure 22. Structures were assigned to these peaks solely on a statistical basis, and in reality, each peak may well represent a mixture of more than one isomer. The configuration shown for each peak in the chromatogram, however, is thought to be the major contributor to that peak. The position of the major component in Convalex 10, peak A, coincides in retention data with the all-meta isomer in the OS 124 fluid, peak A. However, the two minor components, peaks D and E, do not agree with either of the other two OS 124 components. Table 31 shows the relative distribution of the major components found in five OS 124 and two Convalex 10 fluids.

The effect of using the same lot of Convalex 10 oil in three different pumps for approximately 3 months is shown in Figure 23. These curves represent light-end samples, that is, samples collected with a room-temperature baffle and a -75°F collection plate in order to amplify the presence of light-end degradation products. Note that the light end, peak D, in the original polyphenyl ether has disappeared from all samples. The smaller peak, F, is presumably a non-polyphenyl ether light end. The areas (Table 21) of the F peaks in Figure 23 relative to the areas of original main peaks are 60 and 22% for runs 55 and 58 with oil used 3 months; these peaks can be attributed to this original impurity.

An analysis made by Monsanto Company on Convalex 10 used in Station 3 for 2 years is shown in Table 32. This oil was giving very high backstreaming values, presumably because of catalytic decomposition. This analysis is completely different from that on the new Convalex 10. Specifically, the value of the major component, A, is on the same order of 60% as in the OS 124 samples (Table 31). One explanation is that in use the Convalex 10

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is isomerizing and changing towards the OS 124 composition. This phenomenon was also noted in the gas chromatography curves from earlier work (ref. 28).

6. Effect of Adding DC 704 to DC 705

The effect of the addition of light-end components was evaluated by using DC 705 containing 1% DC 704 (Table 33). The DC 704 did not increase the backstreaming (runs 232, 239, and 245). Gas-chromatographic techniques, which evolved along with the work below, showed that the deposits contained from 6.8 to 10.6 wt. % DC 704 (Table 34). A value of 10% is to be expected from an equilibrium vaporization process that takes place at 200°C, where the vapor pressure of DC 704 and DC 705 are 3×10^{-1} and 4×10^{-2} torr, respectively (Table 7).

A check on the ultraviolet absorbance method of analysis was seen when the gas-chromatographic data (Figure 24) were converted to weights of DC 705 and DC 704 (Table 35). The agreement in two of three runs (runs 232 and 234) is considered an excellent independent verification of the analytical method. Time was not available to refine the method by tracer techniques. The one disagreement (run 239) is perhaps a bad sample, since there is considerable strange material coming out on top of the benzene and before the DC 704 (Figure 24, sample 239). A small amount of strange light-end material is found in every one of these backstreaming samples (peak A, Figure 24).

7. Effect of Side-Stream Stripper

One run was performed with a side-stream stripper connected to the oil drain port to test its efficiency in removing the DC 704 from the DC 705 (Table 36). The maximum degree of concentration obtained here was only 1.95/1.26, or 1.6 times, whereas the theoretical equilibrium vapor pressure ratio at 275° C is approximately 4 (Table 7). Presumably, more experience is required to obtain satisfactory performance of this purifier. Under high vacuum the vaporization process is more of a flash vaporization than an equilibrium process. However, the process above the

alembic should tend to approach an equilibrium vaporization.

E. Baffle Performance

1. Optimum Conditions

· The overall performance of the baffles tested under optimum conditions during the course of this program can be summarized as follows. The right-angle elbow and BC-61[*] baffles gave the same low value of 1×10^{-4} mg/cm^2, whereas the HN-6[*] was approximately 3 to 6×10^{-4} mg/cm^2, and the Cryosorb was 10×10^{-4} mg/cm^2. Under operating conditions that are less than optimum, the right-angle elbow gives a much better overall performance. The comparisons given below apply only to the tests under standard and optimum conditions.

A direct comparison of a BC-61 baffle was made with both HN-6 and Cryosorb traps in order to ensure that the differences were due only to the trap (Tables 37 and 38). The HN-6 consistently gave backstreaming values of 7 to 14×10^{-4} mg/cm^2 (runs 216 through 238) compared with 0.9×10^{-4} mg/cm^2 (run 210) when replaced by the BC-61. It is interesting to note that oil migration along the warm sides of HN-6 (run 243) was not pronounced. The values of HN-6 with cold sides are larger than the corresponding value of 5.2×10^{-4} mg/cm^2 with warm sides (run 205). The values for Cryosorb are 8 and 13×10^{-4} mg/cm^2, compared with 2 to 3×10^{-4} mg/cm^2 for the BC-61. The latter values are higher than the optimum value of 1×10^{-4} mg/cm^2, which is usually obtained with the BC-61 baffle.

The large backstreaming values obtained with the Cryosorb baffle were quite unexpected, since this baffle appears to be exceptionally well-designed. Because of these results, the baffle construction was carefully examined. The large values appear to be due to revaporization of oil from the ends of the plates that cover the central cooled baffle section located directly above the pump opening. The bottom plate is always

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[*]Baffle was operated with cooled walls

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covered with a massive amount of oil. The periphery cover
plates, both top and bottom, can presumably run warm. Conse-
quently, the oil on the bottom cover plate can flow around edges
and see the edges of the top plate. Since the latter oil is
warm, it can backstream through the baffle. This explanation
should be evaluated by operating a baffle without the shield
plates over and under the center baffle to determine whether an
optimum performance of 1×10^{-4} mg/cm^2 or less can be obtained.

2. Oil Migration

The major cause of poor performance of the traps under less
than optimum conditions was undoubtedly due to what can be
termed oil migration. One definition of oil migration is move-
ment of oil along relatively warm surfaces by revaporization or
by wetting. Revaporization does not require a continuous path
and is a more usual cause of migration than that due to surface
wetting. This phenomenon is discussed in detail in Section IVF.
The data are summarized into Table 39 to illustrate this pheno-
memon as related to baffles only under essentially optimum con-
ditions.

The right-angle elbow and BC-61 baffles with pentaphenyl
ether give essentially the same increase in deposit weight be-
cause of warm sides, the values being 5×10^{-4} mg/cm^2 for runs
147 and 205 and 1.9×10^{-4} mg/cm^2 for run 109. There is very
little difference in the performance of the HN-6 with and with-
out cooled sides; the values are 10 and 7×10^{-4} mg/cm^2, res-
pectively. Fundamentally, migration should be rate dependent;
however, the rate values show no apparent correlation.

One determination was made on a BC-61 baffle with DC 705
but without a bakeout of the collection plate prior to starting
the run. Comparison of runs 108 and 98 shows that the deposit
increased from 6.5 to 10×10^{-4} mg/cm^2 when cold and warm sides,
respectively, were used.

3. Structural Integrity

Structural defects were found in the first HN-6 baffle

tested. Final performance tests on Station 4 with the BC-61 baffle from Station 1 indicated that this BC-61 was also defective. These small leaks (at system pressures of 1×10^{-8} torr) are extremely difficult to detect. If liquid nitrogen coolant had been used in the baffles, there would have been no effect on contamination level since backstreaming was not affected by small pulsed leaks on the fine side. Organic refrigerants, as employed here, can lead to problems since such compounds are unstable at the temperatures in the boilers.

In Station 1, which employed a probably defective BC-61 baffle for over 1 year on a pump using pentaphenyl ether, optimum backstreaming values of 1×10^{-4} mg/cm^2 were obtained. Station 3 with a defective HN-6 was found to give a value of 10×10^{-4} mg/cm^2, which in final tests was the optimum value for this trap. It can be concluded that these defects and the small leaks of methanol into the system had no effect on the backstreaming values. High backstreaming values obtained in Station 3 during some of these tests due to catalytic effects are discussed later.

F. Operating Procedures

A list of standard operating conditions has been presented (Table 18). The amount of backstreaming oil is directly related to operating procedure, and as such, these procedures constitute an important part of the results.

1. Conditioning

Conditioning is an indefinite term and is used here to describe the operation of a system for some period prior to its actual use to allow the contaminants present to purge themselves. It is often considered good practice to allow a system to idle when it is not being used for specific tests in order to improve or maintain the condition of the oil.

The data (Table 40) show the decrease in backstreaming during a normal conditioning period. This conditioning period

is related to the cleanliness of both the system and the oil,
and although it is a well known phenomenon, it is not well char-
acterized. Throughout the program (with the exceptions dis-
cussed below), the first few runs produced large backstreaming
values that were time dependent. These data are not presented
since there are usually other factors that render analysis dif-
ficult. Obviously, various operating procedures can cause a
system to become and remain "unconditioned."

In the beginning of the program the four clean and con-
ditioned systems from a previous program (ref. 28) gave large
backstreaming values. These systems refused to condition for
an extended period. Although new test heads were used in some
stations and may have contributed to the excessive backstreaming
contamination, the change to a $-75°F$ cooling system combined
with excessive moisture condensation on cold test heads during
sampling was the major cause. This is discussed below together
with the failure of systems to condition due to catalytic oil
degradation.

The data from the previous work (ref. 28 and 29) that show
the conditioning phenomena are confined to Stations 4 and 5, which
employed right-angle elbow baffles, in order to eliminate oil
migration effect. Much of the data, although obtained with
room-temperature baffles and collection plates, definitely show
the effect of a conditioning period. DC 705 in Station 4 de-
creased from 30×10^{-6} mg/cm^2-min after 168 hr (31 days) of
use to 0.1×10^{-6} mg/cm^2-min in 255 hr (49 days) and then re-
mained constant. OS 124 decreased from 0.4×10^{-6} mg/cm^2-min
at 193 hr to 0.02×10^{-6} mg/cm^2-min in 360 hr, whereas Conva-
lex 10 was 0.04×10^{-6} mg/cm^2-min at 408 hr and continuously
decreased to 0.003×10^{-6} mg/cm^2-min. The latter decrease may
have been under optimum conditions and rate independent.

Recent work with Convalex 10 in Station 3 gave 5×10^{-4}
mg/cm^2 in the first run with warm baffle walls and 0.9×10^{-4}
mg/cm^2 in the second run with cold baffle walls. The better
performance for Convalex 10 is to be expected since this material

is subjected to a proprietary treatment for conditioning. One run (Station 5, last run) with a fresh charge of OS 124 showed that there is no essential difference in the two pentaphenyl ethers after conditioning. The long conditioning period of 2 months used for OS 124 in this run was due to press of other work and presumably a shorter period on the order of two weeks would be satisfactory.

The original DC 705 oil was obtained as XF 4770, and its experimental production may be responsible for its pronounced backstreaming and longer conditioning period than the pentaphenyl ether. The latest batch of DC 705 used in Station 2 showed absolutely no need of a conditioning period and gave 1×10^{-4} mg/cm^2 on its first test run. This oil charge was made up with 1% DC 704. However, the same most recent lot of DC 705 when used in Station 4 only conditioned below 10×10^{-4} mg/cm^2 in the last three runs (run 237, Table 27).

The actual factors related to conditioning were not experimentally demonstrated. It does appear that the pump, oil, and baffle combinations tested here should condition to optimum backstreaming values in a matter of 2 to 4 weeks. If a system does not condition within 2 weeks, the best procedure is to clean and recharge the system with fresh oil. Obviously, any one of many factors discussed below can prevent conditioning.

2. Data on Start-up Heat Input

Data (Table 41, runs 93, 95, and 96) on Station 5 using Convalex 10 without bakeout of the collection plate at the start of a run indicated the start-up deposit was minimal when 50% of rated heat input was used for 1-1/2 hr in starting up the diffusion pump. An adequate bakeout procedure should remove all the oil deposit caused by start-up effects. While even after such bakeouts measurable deposits were present, start-up with 50% of rated heat input was adopted as a standard procedure. This involved very little extra effort, and the pump performance, as indicated by the system pressure, was not noticeably affected.

A minimum deposit of 2×10^{-4} mg/cm^2 (Table 41, Runs 93 and 96) was obtained with 50% rated heat during startup and 3×10^{-4} and 4×10^{-4} mg/cm^2 for 25 and 100% rated heat (runs 95, 88, and 89) on startup. The data for run 92 were inconsistent; with two startups at 100% heat input and with the oil exposed to 10^{-2} torr on shutdown, the deposit weight did not double. If the effect is related to the amount of dissolved gas in the oil, this inconsistency could be due to the exposure to the low fore pressure between start ups.

The results for Station 1, with 50% rated heat, showed the same type of effect in three runs (runs 103, 117, and 152) where values of 1 to 2×10^{-4} mg/cm^2 were obtained compared with 4×10^{-4} mg/cm^2 for 100% rated heat (run 100). The effect of oil migration along the uncooled sides of the baffle (runs 100, 103, and 113) may be partially responsible for the differences, although oil migration effects are minimal for pentaphenyl ether.

The backstreaming values for DC 705 in Station 2 were higher than those for pentaphenyl ether, and there is no apparent effect of heat input. The one run with three startups (run 112) and intermediate exposure to 700 torr nitrogen pressure showed absolutely no effect. These data, with the exception of run 153, were uncorrected and are probably high by 1 to 2×10^{-4} mg/cm^2 because of the analytical blank.

It can be concluded that DC 705 gives a slightly higher deposit on startup than pentaphenyl ether. Pentaphenyl ether gives a minimum value with 50% heat input on startup. The data for multiple startup indicated no increase in the amount of deposit; this is an inconsistency, since the multiple startups with air exposure are essentially equivalent to opening the system for sample removal except for the solvent wash treatment. The larger startup deposits for DC 705 may be related to the greater amount of light ends in the oil, as shown by the runs without wall cooling. (runs 98 and 101).

3. Shutdown Procedure

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In the initial runs of this program using -75°F cooling with clean and conditioned systems backstreaming values 10 times greater than those in the original study were obtained (ref. 28). During the first 24 to 48 hr of these runs, very sharp and regular pressure pulses occurred every few seconds. The pulses went into the 10^{-7} torr region, whereas the average system pressure (room temperature) was in the 10^{-8} torr region. These pulses decreased in period and intensity with run time. This phenomenon was almost entirely absent in systems with the right-angle elbow. This type of pulsing could only be eliminated by allowing the baffle to reach room temperature, flooding the system with dry nitrogen, rapidly removing the sample, and starting up. Our previous method of flooding with nitrogen, removing the collection plate, and then rinsing off the plate while still cold did not prevent some internal ice formation. Later, the low temperature plus moisture was the entire cause, and appreciable exposure to ambient air with the system at room temperature did not give this effect. Optimum backstreaming deposits of 1×10^{-4} mg/cm^2 were readily obtainable with ordinary precautions even with appreciable exposure to ambient air if sampling was preceded by an 18-hr warm-up period.

This type of pulsing phenomenon is readily distinquishable from that due to leaks, catalytic degradation, light ends, etc., by the rapid decrease in the pulse period and intensity over a run period of a few hours.

4. Bakeout

In order to eliminate start-up effects, standard conditions were adopted that involved a bakeout period of 18 hr with atmospheric steam at 212°F. The data (Table 42) indicated that there is usually a deposit even at zero run time after an 18-hr bakeout at 212°F. The values are close to 1×10^{-4} mg/cm^2 for pentaphenyl ether (run 139) and 2×10^{-4} mg/cm^2 for DC 705 (run 137). A bakeout temperature of 470°F reduced pentaphenyl ether to essentially zero (run 151) whereas the value for DC 705 remained

unchanged at 2×10^{-4} mg/cm^2 (runs 142 and 143).

The values for the deposits after bakeout at 212°F for both pentaphenyl ether and DC 705 appear to be relatively independent of run time; they varied from 0 to 293 hr. The values of pentaphenyl ether were close to 1×10^{-4} mg/cm^2, whereas DC 705 values were close to 2×10^{-4} mg/cm^2. Variations such as in run 165 (Station 5), which gives a net value 0.3×10^{-4} mg/cm^2, are to be expected because of the accuracy of the analytical method. By taking these variations into account, the bakeout procedure of 212°F is obviously lowering the deposit for pentaphenyl ether from $> 1 \times 10^{-4}$ mg/cm^2 (runs 114 and 152) and for DC 705 from 6×10^{-4} mg/cm^2 (runs 104, 107, and 108). However, one experiment (Station 4) with a seeded plate containing a massive deposit of 25×10^{-4} mg/cm^2 contained 20×10^{-4} mg/cm^2 after baking out at 212°F for 18 hr. The vapor pressure of DC 705 at 212°F is $\sim 10^{-4}$ torr; thus the oil should have vaporized in 18 hr. Obviously, the failure to vaporize any appreciable amount in this case must be due to an oil vapor pressure considerably under the equilibrium value (superheating phenomena).

It can be concluded that a bakeout of 212°F usually lowers the deposits of pentaphenyl ether and DC 705 to 1 and 2×10^{-4} mg/cm^2, respectively. Bakeout at 212°F is not a reliable procedure, however, since conditions are ideal for the superheated liquid to exist for extended periods. It appears that the 1 and 2×10^{-4} mg/cm^2 (1 to 2 monolayers) of pentaphenyl ether and DC 705, respectively, are not removed at 212°F and that such monolayers are removed at 470°F in the case of pentaphenyl ether and not DC 705. These data are generally uncorrected for analytical blank in the case of DC 705 and therefore subject to a slight uncertainity.

5. Cooling of Baffle Walls

The effect of oil migration, that is, the travel of oil vapor along the warm walls of the baffles, is shown in Tables 43 and 44. Cooling the sides of the BC-61 baffle reduced the deposit

from 2×10^{-4} mg/cm^2 (runs 103 and 109) to 1×10^{-4} mg/cm^2 (runs 117, 121, 152, 160, 202, and 208) for pentaphenyl ether and from 18 to 19×10^{-4} mg/cm^2 (runs 98 and 101) to 6.5×10^{-4} mg/cm^2 (runs 108, 149, 161, and 181) for DC 705. There is no appreciable effect of side cooling on the HN-6 baffle when pentaphenyl ether is used; 7 to 10×10^{-4} mg/cm^2 is obtained in any case (runs 243, 233, and 238). It does not appear that this migration effect is directly related to the amount of light ends present, since a light-end run with pentaphenyl ether gave 44×10^{-4} mg/cm^2 (run 146) and a light-end run with DC 705 gave 52×10^{-4} mg/cm^2 (run 234).

It is interesting to observe the value of 5×10^{-4} mg/cm^2 for light ends with the right-angle elbow (run 147). This value is very similar to the value obtained with BC-61 when warm sides were used (runs 100, 103, 109, and 113). A light end run with a right-angle elbow with room-temperature walls should be equivalent to a run with a baffle with room-temperature walls and is a measure of oil migration. The right-angle elbow should permit a much greater amount of oil migration if the mechanism is primarily revaporization of oil molecules. Considerably less than 25% of the molecules hit the room-temperature (warm) walls of the BC-61 baffle, compared with 100% for the right-angle elbow.

6. Trap Temperature

By using our standard backstreaming run conditions, Stations 1, 2, 3, and 5 were operated with baffle temperatures of -22 and -75°F (Table 45). There was no apparent change in backstreaming in this temperature range. The variations in the values for Station 2 were due to the variations in the analytical blank. It would appear from these constant deposit values of 1×10^{-4} mg/cm^2, which are independent with time, that the sticking coefficient for pump oil is essentially unity and independent of temperature under these conditions.

Other factors related to trap temperature that occurred should be considered. Station 3 with a HN-6 baffle and Station 4

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with a right-angle elbow gave high, time-dependent deposits when the oil was presumably undergoing catalytic degradation. This phenomenon implies that the sticking coefficient departed appreciably from unity and that the temperature of -75°F was not effective in trapping backstreaming material.

Only short-term tests were performed with liquid nitrogen cooling of the baffle. Several tests were performed on Station 4 with the Cryosorb baffle (Table 46). There was no radical difference in the results with liquid nitrogen (runs 155, 159, 1963) from those of -75°F (runs 231 and 236). The higher values at -75°F (runs 176 and 187) are probably due to contamination following the light-end run (run 168).

7. Leaks

The effect of leaks in the fine and rough side was evaluated (Table 47 and 48). The admission of oxygen into the fine vacuum side of a station employing Convalex 10 had no effect on the amount of backstreaming oil deposit; the value was 0.7×10^{-4} mg/cm^2 over a run period of 290 hr (Table 47). A small leak of air into the fore-line (50μ) had no effect, but a large fore-line leak (run 222) with a fore-line pressure of 0.4 torr caused pulsed pressure readings in the fine vacuum and an oil deposit of 19×10^{-4} mg/cm^2 during a run of 146 hr (Table 48). The oil deposit is probably due to unstable pumping action, which caused some of the condensed mist in the vapor space around the jet stack to be ejected through the baffle.

8. Fore Pressure and Fore Pump

Optimum backstreaming values of 1×10^{-4} mg/cm^2 can be obtained routinely over extended periods with fore pressures of 10^{-2} torr. No contamination from the mechanical system was detected. No specific tests were made for this oil; however, experience with a similar oil indicated such contamination would be accompanied by unsaturated hydrocarbons, which, in turn, would result in large and inconsistent ultraviolet values. The vacuum

systems were normally operated with small fore-line leaks and ballasted mechanical pumps and should be completely free of oil migration.

The use of a diffusion pump in the fore-line is often recommended for two reasons: prevention of contamination by mechanical pump oil, and removal of light-end contamination from the main diffusion-pump oil. The latter was tested for Station 3 before it was known the HN-6 baffle was defective. The tests showed a direct influence of fore-line pressure on the period of hydrogen bursts. However, no correlation with backstreaming measurements was attempted.

G. Mass Spectrometer Studies

1. Gaseous Backstreaming

The use of a mass spectrometer with gas injection is a well-known technique for studying the operation of diffusion pumps. This technique can be developed sufficiently to permit rapid and sensitive measurements of backstreaming phenomena. Here, backstreaming is used to signify the movement of any material, including gases such as hydrogen, backwards through the pump. It is more common to consider the effect with hydrogen as a measure of pump efficiency rather than a backstreaming phenomenon. The objective of these experiments was to study hydrogen backstreaming and attempt to relate it to the mechanism involved in the backstreaming of the heavier components.

There are two main mechanisms by which material can move backward through a diffusion pump. One involves direct flow through the vapor jet countercurrent to the gases being pumped. The second involves solution of the gases in the condensing pump oil and reissue through the jets together with the oil vapor. This material then curves around and up due to the random motion that exists in these jet streams. The classical form of a fractionating diffusion pump employs a compartmented boiler so that the vaporizing oil furnishes vapor from lower to higher jets

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consecutively. Some recently designed pumps subject the condensed oil to a hot-wall section prior to its reentry into the boiler. This type of operation is presumed to remove the light ends before the liquid oil reaches the boiler.

The modern oils with excellent stability and very small amounts of light ends from oil degradation have decreased the need for the fractionating design. Such designs are usually based on relatively short-term tests and the results can be quite different in long-term usage. It is probably that continuous liquid nitrogen cooling of the baffle could delay the appearance of light ends by freezing them out throughout any extended test period.

2. Mechanism Studies

The rate of appearance of gases in the test head, as monitored by the mass spectrometer and an oscilloscope, should depend upon the mechanism of backstreaming and the point of gas injection. Thus, gas diffusion through the top jet itself should be practically instantaneous, whereas backstreaming via solution in the oil and out with the vapor stream should involve appreciable time, probably seconds. The experimental problem is complicated by the nature of gases, since different species have different degrees of oil solubilities, and since there is an interplay of concentration streams due to the different fractionating processes in the pump as well as effects due to the location of the leak.

The purpose of the initial experiments was to observe whether any significant differences could be observed when hydrogen, helium, or air was injected into the vapors just below the top jet or into the liquid oil outside of the bottom of the jet stack. In all of these experiments, the gas entered in pulses that were extremely reproducible for a given leak setting and a given gas. The usual phenomenon consisted of a quiescent period of 3 to 10 sec with a constant low gas (sometimes zero) concentration followed by a large pulse and then series of gradually diminishing

pulses at fairly uniform periods. No visual differences in the oscillographs could be detected in any series of experiments. The series of diminishing peaks indicates the absence of solubility effects that would not only change the period but also smooth them out.

These experiments were conducted at high leak rates so that they could be observed on the oscilloscope. Consequently, the effects at lower pressures could be completely masked by the magnitude of the leak. A more satisfactory scheme would involve the use of a carrier gas containing several components of interest. The study of relative changes would eliminate the time element and permit differences to be detected at low partial pressures.

The values of the overall compressibility factors for selected runs of helium and hydrogen are given in Table 49. Arbitrary judgement is involved in selecting the average value since most of the readings are pulsing to some extent. The values cited are presumably within 75% of the average value. The data are presently too limited for any definitive conclusions to be drawn. However, a plot (Figure 25) of the compressibility factors* versus the rates (abcissa changed to give rough equivalence in leak rates) show similar changes for both helium and hydrogen except for the initial value of the hydrogen at leak 3 (into the liquid oil at bottom outside jet stack).

3. Partial Pressure Analyses versus Backstreaming

Backstreaming could not be predicted by monitoring the high vacuum for heavy mass peaks. This is to be expected because of the random and nonhomogeneous nature of backstreaming in baffled systems. Moreover, contaminated surfaces would not be expected to consistently exert equilibrium vapor pressure, as the bakeout data has shown.

Mass spectrometry curves are given for Stations 3 and 5 with polyphenyl ether and -75°F baffles with either room-temperature

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*Also called ultimate pressure ratio.

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or -75°F collection plates (Figures 21, C, D, E, and 26). All of the peaks above m/e = 50 are in the low 10^{-11} amp region and those above m/e = 60 are in the 10^{-12} amp region. Since -75°F baffle has a pronounced effect on peak height and since benzene has a vapor pressure of 10^{-4} at -75°F, these peaks must all be due to high-molecular-weight species. The measured sensitivities for benzene, 0.00001 and 0.006 amp/torr (Table 23), for small and large magnets, respectively, indicated that an appreciable amount of benzene molecules must be present before they show up on the partial pressure analyzer.

The values for backstreaming deposit and light ends for these stations were extremely high (17 to 300 x 10^{-4} mg/cm^2) compared with our usual values of 1 and 5 x 10^{-4} mg/cm^2, for these baffles. It is not easy to make obvious comparisons since the backstreaming here is a time-dependent value. It does appear, however, that the large deposits and the high rate (1 x 10^{-6} mg/cm^2-min) are below the limiting sensitivity range of the partial pressure analyzer.

No measurements were made on DC 705 since it is doubtful whether effective bakeout procedures can be employed to clean up the mass spectrometer tube or the test head. Bakeout data that indicate contamination even after bakeout at 212 and 470°F have been presented. In a parallel program (ref. 30) a Schueman gauge, which has large metal surfaces, was exposed to DC 705 and then baked for 7 days at 500°C but it still showed surface contamination. Hablanian has presented mass-spectrometric data (ref. 20) that show numerous peak values in the 10^{-11} to 10^{-10} amp range for m/e values of 65 to 80 for DC 705 when an unbaked system with liquid nitrogen trapping is used. His data were presumably obtained with the small magnet and with a benzene sensitivity of 0.0001 amp/torr (by our measurements, Table 23). The backstreaming value in Hablanian's 524-hr run was 0.07 x 10^{-6} mg/cm^2-min, which is equal to 22 x 10^{-4} mg/cm^2. Bryant and Gosselin in their original work (ref. 4, Figure 15), presumably using a small magnet and a small DC 705 system and 500°C bakeout,

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show values of m/e = 78 with room-temperature zeolite and cold zeolite trapping. These values are compared with our mass spectrometer readings for pentaphenyl ether in Table 50. During the mass-spectrometric tests, backstreaming values of 1×10^{-6} mg/cm^2-min in 100-hr runs (60×10^{-4} mg/cm^2) were obtained. These very large values are suspect. They may be due either to the air leaks or more probably to upsets from experimental manipulations.

It can be concluded that the mass spectrometer gives a lower reading for m/e = 78 on pentaphenyl ether than the published DC 705 data, even when considerable backstreaming deposits have occurred.

Recently, Craig and Harden (ref. 9) obtained complete spectral data on many pump oils, including pentaphenyl ether and DC 705, at 170°C by using a high-resolution mass spectrometer. Their data show that with pentaphenyl ether, the frequency of mass 77 is 80% of the main mass peak of 446, whereas with DC 705 the masses below 100 are only 5% of the main peak at 531. These data indicate DC 705 contamination, as evidenced by the mass 78 $\pm$ 3 peaks, should be 1/16 as pronounced as with pentaphenyl ether for the same amount of contamination.

4. Sensitivity Measurements

A series of mass-spectrometric sensitivity measurements was made for helium over a period of several months (Table 51). The results are very reproducible on each day (within 20%) except when the leak rate is so low that the system pressure differences do not give a reliable reading of the leak gas pressure. However, the sensitivity changed radically from day to day. In later experiments, reproducible sensitivities between 0.011 and 0.019 amp/torr were obtained.

Sensitivities were also determined for a number of other gases over a period of months and for various experimental conditions (Table 52). Sensitivities for nitrogen and oxygen were determined by both direct and indirect comparison with the

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Bayard-Alpert gauge. Values for water, methanol, and benzene were determined by indirect comparison. Sensitivities for nitrogen varied from 0.045 to 0.17 amp/torr, for oxygen from 0.008 to 0.13 amp/torr, and for water from 0.07 to 0.28 amp/torr. In general, the values for the small magnet are lower than those for the large magnet. Comparison of sensitivities measured on the same day showed that changing the magnet changes sensitivity by a factor of two for masses 28 and 32. The effect was even greater for benzene (mass 78), for which the sensitivity is 0.00001 amp/torr with the small magnet and 0.003 to 0.022 amp/torr with the large magnet.

In view of the importance that has been attached to the presence of $m/e = 78$ measurements in diffusion pump systems, data for a known mixture of benzene in air are given (Figure 27 and Table 51). The mass-spectrometric values were individually surveyed by manually controlling the actual m/e values, and only the values shown were surveyed. The values were checked several times over a period of several days; sensitivities varied from 0.003 to 0.016 amp/torr (Table 52). The agreement with API values seems quite good. The most important result of the data is the low sensitivity values obtained.

V. Summary of Results

1. Under optimum conditions, no detectable deposit ($<1 \times 10^{-4}$ mg/cm^2, <1 monolayer) is formed after the bakeout period in runs up to 544 hr.

2. Optimum conditions with no detectable deposit increase could be obtained routinely with either pump, all 3 oils, and 2 out of 4 baffles.

3. A monolayer deposited during startup and bakeout is not removed by 212°F bakeout but is removed by the solvent wash during analysis.

4. The ultraviolet analytical method was demonstrated to be definitive to 1 monolayer (1×10^{-4} mg/cm^2) by check methods involving seeding, weight, and gas chromatography.

5. A gas-chromatographic method developed for DC 705 is suitable for 0.1 monolayers and for defining components such as DC 704 down to 1%.

6. No differences were found in the performance of the two pumps, CVC-PMC-1440A and NRC HS6-1500.

7. The performance of three oils, DC 705, Convalex 10, and OS 124, was the same under optimum conditions.

8. DC 705 showed no visual signs of degradation in several years of continuous use.

9. Pentaphenyl ether formed dark deposits on external surfaces after several years, but the slight degree of these deposits would permit many more years of operation.

10. Both DC 705 and pentaphenyl ether appeared to undergo auto-catalytic degradation, which was accompanied by large back-streaming values. The cause and cure are not known. The only effective solution is thorough cleaning and oil change.

11. The effect of extensive air leakage on the stability of pentaphenyl ether was found to be transient and rapidly

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self-corrective upon removal of the leak.

12. The phenomenon of explosive revaporization can be used to explain the usually superior performance of pentaphenyl ether versus DC 705 and right-angle elbow versus BC-61.

13. There was no performance difference found between the two pentaphenyl ether fluids, Convalex 10 and OS 124. After extensive use Convalex 10 gave a gas chromatogram similar to that of OS 124.

14. The optimum performance of DC 705 with the added light-end component DC 704 was 1×10^{-4} mg/cm^2, which is the same without this component. A side-stream stripper had no apparent effect on removing this light end.

15. Under optimum conditions, the two baffles, right-angle elbow and BC-61, gave the same low value of 1×10^{-4} mg/cm^2. HN-6 gave 6×10^{-4} mg/cm^2, and the Cryosorb baffle, 10×10^{-4} mg/cm^2.

16. A major cause of poor baffle performance is movement of oil along relatively warm walls by vaporization or wetting.

17. Oil migration occurs along the warm walls of the BC-61 baffle; the values are 2 to 4×10^{-4} mg/cm^2 over the optimum for pentaphenyl ether and approximately 12×10^{-4} mg/cm^2 for DC 705. The oils have essentially the same amount of light ends, 44 and 52×10^{-4} mg/cm^2, respectively.

18. For optimum performance the BC-61 baffle requires wall cooling to eliminate oil migration.

19. Under most conditions, right-angle elbow gave better performance than the HN-6, probably because it offers less opportunity for explosive revaporization. The right-angle elbow with warm walls gave the same value as the BC-61 with warm walls, $\sim 5 \times 10^{-4}$ mg/cm^2. However, the equivalent operation, BC-61 with no cooling, resulted in values of 20 to 100×10^{-4} mg/cm^2.

20. The HN-6 baffle showed no pronounced oil migration effects, and its backstreaming with and without cooled sides was greater than the BC-61 with room-temperature walls: 7 to 12×10^{-4} mg/cm^2 compared with 2 to 5×10^{-4} mg/cm^2, respectively.

21. The Cryosorb baffle gave the poorest performance (10×10^{-4} mg/cm^2) in these tests, presumably because of vaporization from the warm periphery of the bottom cover plate, which is over the central liquid nitrogen baffle.

22. A conditioning period of 2 to 4 weeks was often required for a system to purge itself of contamination and give optimum backstreaming values of 1×10^{-4} mg/cm^2. In two instances, fresh DC 705 and Convalex 10 did not require a conditioning period.

23. A programmed rate of 50 and 100% of rated heat input was usually found to give minimum backstreaming on startup for pentaphenyl ether.

24. Multiple startups with intermediate exposure to atmospheric air gave the same value as a single startup. This anomalous result cannot be explained.

25. The presence of excessive moisture due to sampling with the baffle still cold resulted in large backstreaming values. The pulse phenomenon in gauge readings indicated that explosive vaporization was probably involved.

26. A bakeout procedure of 212°F will usually limit startup deposits to 1×10^{-4} mg/cm^2. The absorbed material can be removed at 470°F in the case of pentaphenyl ether but not DC 705. With DC 705, massive oil deposits of 25×10^{-4} mg/cm^2 produced by seeding and subjected to 18 hr at 212°F, resulted in a residual of 20×10^{-4} mg/cm^2.

27. Trap temperatures varying from -27° to -75°F gave the same optimum performance values.

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28. The effect of leaks in the fine side, such as air, oxygen, and moisture, with system pressures from 10^{-6} to 10^{-8} torr (P_{LN_2}) resulted in the same optimum value of 1×10^{-4} mg/cm^2 as under standard conditions. Mass-spectrometric studies with pentaphenyl ether showed a transient presence of degradation products.

29. Leaks in the fore line, with a fore pressure of 0.4 torr, increased the backstreaming values to 19×10^{-4} mg/cm^2. Pulsing fine-pressure readings indicated that unstable pumping action of the jets was responsible.

30. The use of an untrapped fore line with leaks at 1 to 30×10^{-3} torr and a ballasted mechanical pump gave routine optimum backstreaming values of 1×10^{-4} mg/cm^2. There was no evidence of contamination by mechanical pump oil.

31. Use of low 10^{-5} (P_{RT}) fore pressures had no effect on a station that gave high backstreaming values of 19×10^{-4} mg/cm^2. These high values were probably due to autocatylic degradation as shown by the periodic hydrogen bursts that occurred.

32. A study of gaseous backstreaming with mass spectrometry by employing gas injections of hydrogen, helium, and air in various locations within the pump showed no significant differences.

33. Mass-spectrometric measurements indicated no backstreaming contamination with pentaphenyl ether systems. The mass-spectrometric sensitivity values for different gases varied from day to day, up to 10 times, in an unpredictable manner. The values for benzene were 0.00001 and 0.003 amp/torr for the small and large magnets, respectively.

PART B. BACKSTREAMING FROM TURBOMOLECULAR PUMPS

I. INTRODUCTION

The recently developed turbomolecular vacuum pump (ref. 3) is receiving considerable attention from workers in the high-vacuum field. Invariably, the users are interested in the ability of this untrapped pump to operate without introducing contamination in the vacuum space by backstreaming of the lubrication oil. Consequently, the objective of the second phase of this program was to evaluate the backstreaming contamination in these devices as received and, after bakeout, to determine the influence of shut-down and start-up procedures.

The results indicate that deposits are less than 1 monolayer. However, considerable amounts of vapor-phase heavy ends were found in the vacuum chamber by mass-spectrometric measurements. The measured values of the deposits are not as well characterized as in the diffusion-pump studies. Oil degradation products from the lubricating process that are believed to be present influenced the ultraviolet measurements in a variable and unpredictable manner and necessitated the development of a gas-chromatographic technique.

II. TECHNICAL DISCUSSION

The operation of the turbomolecular pump is similar to that of a turbocompressor, in which the gas is compressed by alternate rows of rotor and stator sections. Pumping action is effected when the peripheral speed is faster than the gaseous molecular velocities. By the correct choice of blade angles, this pump is able to utilize clearances on the order of 1 mm between the stator and rotor. The feasibility of using these large clearances has made it possible to design a practical

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pump. Molecular pumps with a 0.02 mm clearance were described fifty years ago (ref. 17).

All molecular pumps are reputed to give clean vacuums. This should follow since the pumping efficiency increases with the molecular weight of the gas. The reported compression ratios (ref. 3) for the turbomolecular pump are: hydrogen, 250; deuterium, 2400; air, 1.5×10^7; Freon-12, (molecular weight = 158) $>8 \times 10^8$. The pumping speeds, however, vary in the opposite direction: 600, 500, and 250 m^3/hr for hydrogen, air, and Freon-12, respectively, in a pump with a 170-mm-diameter rotor. "Mechanical" contamination by the lubricating oil is avoided by placing the inlet port in the center and pumping symmetrically to either end. Thus the bearings are exposed to the fore-pressure system only. However, on shutdown or startup there is a period of 10 to 20 min, before the turbomolecular pump is up to speed, during which the forepump can lower the pressure below the vapor pressure of the oil. There is also the possibility that the oil will froth at a much higher pressure because of dissolved gases. This situation is avoided by maintaining the fore-line pressure above 1 torr until the turbomolecular pump is up to speed.

III. EXPERIMENTAL WORK

A. Test Stations

The turbomolecular pumps were supplied by the Welch Scientific Company as a complete unit (Table 54 and Figures 28 and 29). These pumps were used without any failsafe instrumentation. The perforated insert for the pump inlet was not used. The fore line was provided with a "christmas tree" (same as fore line in diffusion pump system) to allow gauging and pressure control on startup. A different test head was employed (Figure 30) that combined all of the functions of collection plate and test spool used in the diffusion-pump test stations. The test head allowed the use of dry, double O-ring seals throughout. In the last runs, one

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turbomolecular station was supplied with a BC-61 baffle, which was used at room temperature, -75°F, and liquid nitrogen temperature, during the mass-spectrometric tests. Provision was made so that any desired gas could be leaked into the fine or fore side. Control was obtained with Granville Phillips variable leak valves.

B. Procedures

The majority of experimental procedures concerned with oil diffusion pumps is applicable to the work with turbomolecular pumps. The following section describes the significant differences.

1. Standard Conditions

A fundamental phase of the program was to evaluate the turbomolecular in as-received and baked-out conditions for optimum operation. Table 55 lists the recommended run procedure, which should give minimum possibility of contamination due to the volatility of the lubrication oil. Test procedures that were varied for conditions of no bleed, bakeout, or back flush are discussed in Section IVD.

The fore-line pressure is critical to the turbomolecular pumping speed (Table 56). The system pressure decreased immediately when the leak rate was lowered; thus the turbomolecular pump was pumping against the resistance of the leak. We also increased the fore-line leak and noted the fine pressure rise as the leak increased. The critical fore-line pressure appeared to be 1.01 mm Hg. Above this pressure the fine vacuum pressure went to about 8×10^{-4} torr.

2. Analytical Techniques

Techniques very similar to those for DC 705 and pentaphenyl ether were used throughout these studies. The ultraviolet technique, though suitable for unused or bulk turbomolecular oils, proved completely unsatisfactory for the oil deposit and necessitated the development and use of gas-chromatographic techniques.

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3. Solvent Treatment of Collection Plates

This procedure was identical to that used for the diffusion-pump study except isooctane was used as the solvent instead of methanol.

4. Ultraviolet Absorbance

This procedure was identical to that used in the diffusion-pump study except a 2-cm cell path was used instead of the 1-cm path for all but the initial calibration measurements. Calibration data (Table 57) gave a slope of 0.516 in concentration versus absorbance units measured at a wavelength of 252 mμ. The amount of oil was calculated from:

$$W = V \, m \, A.U.$$

W = total weight of oil on collection plate, mg

V = total volume of rinse sample, ml

m = slope of calibration curve 0.516 (2-cm cell) mg of oil/ml/A.U.

A.U. = absorbance unit determined in the Cary model 14 spectrophotometer at 352 mμ wavelength for turbomolecular pump oil dissolved in isooctane.

Table 58 shows the accuracy of the ultraviolet analytical methods by seeding. The last seeding experiment indicated that 2×10^{-4} mg/cm^2 was not detectable by ultraviolet techniques.

5. Gas Chromatography

The use of gas chromatography to analyze the turbomolecular deposit was necessitated by the large and random nature of the ultraviolet analyses on run samples. The technique used for DC 705 was employed for turbomolecular oil except the column and injector port temperatures were 165 and 240°C, respectively.

Fundamental gas-chromatographic data on turbomolecular pump oil referred to hydrocarbon standards is given in Figures 31 and 32 and Tables 59 and 60. These samples were unused oils and were not concentrated. The mixed components present in the oil did not give a good base line. The recovery was only 7.6%, based on sensitivities determined with pure hydrocarbons. In order to relate

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the above and subsequent results obtained with used turbomolecular oil, comparisons were made with unblended turbomolecular stock (Mobil 113 stock). The curves (Figure 33) were identical. The infrared spectra are compared in Figures 34 and 35.

The turbomolecular oil contains DC 200 as an antifoam agent. The curve with added amounts of DC 200 at the standard temperature of 165°C gave no strange peaks. However, the curve at 300°C (Figure 36) showed that all of the peaks from DC 200 came out before C_{16} and thus cannot interfere with the analysis.

The reason for the effort on gas-chromatographic analysis was the large and random nature of both the sample and the analytical blank by ultraviolet, which generally gave impossibly high and/or negative values (Table 61 and 62). These actual ultraviolet samples were used to establish the technique. Emphasis on DC 705/704 analysis by gas chromatography prevented further efforts in known seeded samples.

The standardization values are given in Table 63. The run data are given in Figures 37 through 40 and are compared in Table 64. All values are based on average readings on the two peaks corresponding to C_{16} and C_{17}. The reliability of these values is indicated by the close agreement of the ratio to unity except for run 214B. Note that the standard value is based on the weight of oil. Therefore the values by gas chromatography are 100% relative to the amount of oil and arbitrarily correct the 7.6% recovery (Table 60) to 100%. These data are discussed further in Section IV. At this point, it is sufficient to state that the low values by gas-chromatography are consistent, and the ultraviolet values are not, presumably because of the strange component that shows up as Peak S in Figures 39 and 40.

IV.   RESULTS

A.   Backstreaming Contamination under Standard Conditions

The backstreaming values determined by gas-chromatographic analyses were less than 1×10^{-4} mg/cm^2 (Table 64, runs 201, 214, and 215). The mass-spectographic curves indicated the presence of considerably more heavy mass peaks than were detected in oil-diffusion-pump systems under optimum conditions, presumably because of cracked hydrocarbons resulting from the lubrication process. These results were obtained in baked systems. Time was not available to evaluate in depth the effect of operating procedures on these gaseous contaminants.

B.   Validity of Measurements

The results of gas-chromatographic analyses were very consistent (Table 64) and were probably an accurate measure of the backstreaming deposit to within 1×10^{-4} mg/cm^2. Since they were only developed recently, they were not verified by seeding experiments or by changes in operational procedures to a degree that was desired.

The results of the many measurements by ultraviolet techniques (Tables 61 and 62) were generally inconsistent. The magnitude and variation in the analytical blank cannot occur with turbomolecular oil per se, since the seeding tests (Table 58) showed very high solvent efficiency in removing oil. Furthermore, any of the net deposits of 30×10^{-4} mg/cm^2 and over (runs 189 and 201) should have been visually evident. Notice that analytical blank B is often larger than blank A. A series of five blanks (run 241) was made, and again the second blank showed this strange jump. A series of four blanks (run 242) showed no jump but four consistently high values. The gas chromatograms (Figure 39 and 40) show the strange peak, S, present in blank B and not in the original sample. It is quite probable that this peak material is responsible for the variations. The phenomena of

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this material showing up after numerous and copious rinses in a small 10-ml rinse is not consistent with known material transfer processes. It was demonstrated that the antifoam agent, DC 200, did not interfere with either the ultraviolet or gas-chromatographic technique.

It is probable that the cracked hydrocarbons from the lubrication process are responsible for the anomalous results. In small quantities these unsaturates have large ultraviolet absorbance characteristics. Furthermore, the mass-spectrometric results indicated the presence of numerous mass peaks from 45 to 100. These follow logically from the same source.

The gas-chromatographic analyses (Table 63) were consistent in all but run 214. However, the ratio of C_{16} and C_{17} for only this run (Figure 39) was 0.38, compared with the standard value of ~ 1. This result indicates that the gas chromatogram is in error.

The low recoveries, 2 to 14% relative to the ultraviolet values, are to be expected if the deposits contain unsaturates. The unsaturates can also affect the gas-chromatographic results but the constant C_{16}/C_{17} ratio shows that these components are present in the normal ratio. The 7.6% recovery based on C_{16}, C_{18} and C_{21} (Table 60) is not particularly abnormal in view of the nature of turbomolecular oil. (It should be noted that the recoveries of 2 to 14% in Table 63 for gas chromatography/ultraviolet are corrected to 100% by weight of turbomolecular pump oil; that is, 7.6% recovery by C_{16} is 100% by weight. Based on calibration and the many anomalies of ultraviolet data, gas chromatography is more reliable. Obviously, better correlation can be obtained with additional time.

C. Mass Spectrometer Studies

Station 8 was surveyed by the General Electric partial pressure analyzer (Figure 41)*. In every case, the test head was

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*Peaks were surveyed individually and the absence of a given peak does not necessarily indicate the peak was not present.

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baked out for 18 hr at 212°F. The fore-pressure leak was used on startup. The first survey was made with the small magnet on run 209 at 760 hr. The system pressure was 3×10^{-9} torr (P_{RT}) and 4×10^{-10} torr (P_{LN_2}) with a cold collection plate. The P_{RT} increased to 4×10^{-8} torr when the collection plate was warmed to room temperature. The major component at the water peak, m/e = 18, shows considerably less change, 2×10^{-9} to 3×10^{-9} amp, than the increase in total pressure. The warm plate does show several new peaks around m/e = 30 that could be responsible for this difference. More complete surveys made with the large magnet are also shown in Figure 41 (C and D).

The most interesting feature is the comparison of this survey with that from an oil-diffusion-pump system. The latter shows peaks that are 10 to 100 times smaller. Comparisons of backstreaming values are not definitive in either case. The backstreaming values for the turbomolecular oil by ultraviolet techniques are 10×10^{-4} mg/cm^2, and by analogy with gas chromatography results, are probably 1×10^{-4} mg/cm^2. The backstreaming values for the diffusion pump were very high, $>60 \times 10^{-4}$ mg/cm^2, during the mass spectrometric tests, presumably due to blow-back upsets during the leak tests.

In order to evaluate heavy-end backstreaming from the turbomolecular pump, a BC-61 baffle was added. Experiments with room-temperature and liquid nitrogen baffles are also shown in Figure 41 (F and G). The decrease in peaks due to cooling was uniform over the entire mass spectrum and thus indicative of a contaminated test head and baffle. The baffle did not have cooled sides and had been subjected to a thorough solvent cleaning and baking procedure. If these heavy ends were coming from the pump, all heavy ends above 45 would have been cut off abruptly or at least to a great extent. Another complication is that the DC 200 antifoam agent could have contaminated the test head or mass spectrometer tube and be responsible for the heavy peaks.

The residual gas was analyzed again (Figure 41, H) with a room-temperature baffle, after the entire head, baffle, and pump center had been baked at 212°F for 5 days. The entire spectrum

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as well as the system pressure decreased quite uniformly, indicating again that residual contamination was responsible and that the extensive baking period merely made a fractional improvement. Upon dissembly and inspection of the pump, the rotor and sections of the stator were wet with oil. It is obvious that the bakeout procedure for the inlet and central rotor section was inadequate.

D. Operating Procedures

The results of all the ultraviolet analyses of backstreaming contamination (Tables 61 and 62) were inconsistent and are probably high by manyfold. Only the results on baked systems (Table 64, runs 201, 214, and 215) by gas-chromatographic techniques, which give backstreaming values under 1×10^{-4} mg/cm^2, are believed valid. As a result of the analytical ambiguities, which were only resolved at the end of the program, different operating procedures were not evaluated experimentally.

The experimental procedure shown in Table 55 will minimize backstreaming of lubricating oil on startup. The lubricating oil can backstream due to both its highly volatile nature and more probably due to frothing caused by dissolved gases. Our tests show that with 212°F backout, the oil deposit on the collection plate was minimal. The considerable gaseous contamination was probably due to cracked hydrocarbons in the test head. These were not removed by 212°F bakeout for 1 week. Moreover, oil deposits on the central rotor and parts of the stator were not removed by a 212°F bakeout at the pump inlet and center.

Shut-down procedures involving bleeding of inert gas to the fine vacuum and bringing the system pressure above 5 torr before turning off the rotor should be a routine procedure and should eliminate backstreaming contamination during this operation.

There is considerable doubt that a bakeout at 212°F removes adsorbed unsaturated hydrocarbons, and this should be tested. These components can be very low-molecular-weight materials and can breath out of the oil at relatively high pressure.

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V. RESULTS

1. The backstreaming values determined by gas-chromatographic analyses were less than 1×10^{-4} mg/cm^2. The mass spectrometric curves indicated the presence of heavy mass peaks that are probably unsaturated hydrocarbons resulting from the lubrication process.

2. The gas-chromatographic analyses are believed accurate to 1×10^{-4} mg/cm^2 but have not been verified by seeding experiments. The ultraviolet technique, while demonstrated to be accurate by seeding experiments with unused oil, gave large positive and negative values that were obviously in error. This type of discrepancy can be caused by the presence of unsaturated hydrocarbons. However, the variations in the analytical blanks are not consistent with known material transfer processes.

3. Mass spectrometric studies showed the presence of mass peaks over 60. These peaks, which persist with a liquid nitrogen baffle, are probably due to contaminated surfaces in the test head and not backstreaming from the pump.

4. A prolonged bakeout at 212°F was not sufficient to remove contaminants in the test head. They are probably unsaturated hydrocarbons and strongly adsorbed on the surfaces.

5. A prolonged bakeout at 212°F of the inlet and central rotor section was inadequate to remove residual lubricating oil films.

6. The influence of startup and shutdown procedures on minimizing contamination was overshadowed by surface effects. The use of high fore pressures on startup and an inert gas bleed in the fine side on shutdown should prevent backstreaming of the lubricating oil while the rotor is not up to pumping speed.

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APPENDIX

TABLES AND FIGURES

Table 1

CONTAMINATION LEVELS IN MONOLAYER EQUIVALENTS

---

1. Molecular diameter calculated on basis of face-centered cubic packing (ref. 13):

$$d = 1.329 \times 10^{-8} \ (M/\rho)^{1/3} \ cm$$

   where M = molecular weight
          ρ = density of condensed phase, g/ml.

   a. Polyphenyl ether $d = 0.99 \times 10^{-7} \ cm$

   b. DC 705 $d = 1.09 \times 10^{-7} \ cm$

2. Weight equivalent for a monolayer:

   a. Polyphenyl ether $0.82 \times 10^{-4} \ mg/cm^2$
   b. DC 705 $1.00 \times 10^{-4} \ mg/cm^2$

3. Deposition rate equivalent for a monolayer:

   $$1 \times 10^{-6} \ mg/cm^2 \text{-min} = 1 \ \text{monolayer}/100 \ \text{min}$$

Table 2

REPRESENTATIVE VALUES OF MASSIVE BACKSTREAMING

	Pump	Size, in.	Conditions	Backstreaming $mg \times 10^{-6}/cm^2$-min	Ref.
1a.	NRC HS6-1500 (no cold cap)	6	AVS	20,000	21
1b.	NRC HS6-1500 (no cold cap)	6	Collection surface 10 in. from flange	500	21
1c.	NRC HS6-1500 (cold cap)	6	AVS	400	21
2a.	NRC H32-32000 (cold cap)	32	AVS, fore pressure 10^{-3} torr	660	21
2b.	NRC H32-32000 (cold cap)	32	AVS, fore pressure 10^{-2} torr	13,000	21
3a.	Edwards 203B (no cold cap)	2-1/4	AVS, 250 watts, DC 704	14,000	10
3b.	Edwards 203B (no cold cap)	2-1/4	AVS, 250 watts, Apeizon C	7,500	10
3c.	Edwards 203B (no cold cap)	2-1/4	AVS, 250 watts, DC 705	7,500	10
3d.	Edwards 203B (no cold cap)	2-1/4	AVS, 200 watts, DC 705	4,000	10
3e.	Edwards 203B (no cold cap)	2-1/4	AVS, 300 watts, DC 705	11,000	10

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Table 3

ANALYTICAL TECHNIQUES AVAILABLE
FOR MEASURING TRACE CONTAMINATION

1. Quartz crystal controlled oscillator (ref. 40):

   weight to less than 10^{-12} g/g.

2. Field emission microscope pattern (ref. 33):

   change with adsorption of a monolayer on a clean surface.

3. Field emission ultramicrometer (ref. 43):

   measures 10^{-6} cm, accuracy $1/10^5$ parts.

4. Magnetic flux ultramicrometer (ref. 44):

   measures: actual, 2×10^{-7} cm; theoretical, 2.6×10^{-9} cm.

5. Multiple-beam interferometry (ref. 39):

   measures 10 to 10,000 A, 1 mm^2 surface.

   a. actual measurement on DC 705 (ref. 24):

      masked section, polymerized deposit by electron beam, 50 A, 0.5 A/hr.

   b. commercial instrument (ref. 14):

      measures 10 A to 15,000 A, 4% reproducibility at 120 A.

6. Gas chromatography (ref. 16):

   actual: 10^{-7} g C_{21} full scale;

   theoretical: 2.5×10^{-9} g C_{21} full scale.

7. Optical level (ref. 26):

   measures 10^{-10} cm.

8. Radioactive tracers (ref. 5):

   actual measurements on DC 704, 0.01×10^{-5} mg/cm^2 = 0.1 A.

9. Trace analysis by various methods (ref. 1):

   measures 0.1 ppb.

10. Wettability (ref. 15):

    measured: 30 A mineral oil, 5 A lard oil, 1 A stearic acid.

11. Impurity detection (ref. 42):

    measures ppb in high-purity materials, thermally stimulated currents for insulators.

12. Ellipsometry (ref. 34):

    measures 70 A.

Table 4

SPECIFICATIONS OF MECHANICAL PUMPS

Manufacturer:	The Welch Scientific Company, Skokie, Ill.
Model	Duo-Seal vacuum pump No. 1397B
Speed	13 cfm, free air
Ultimate pressure	10^{-4} torr, with ballast closed (always operate with ballast 3/4 turn open)
Type	two stage
Date Purchased	August 1962

Table 5

SPECIFICATIONS OF DIFFUSION PUMPS

	Fractionating[a]	Nonfractionating[b]
Manufacturer	NRC Equipment Corp. Newton, Mass.	Consolidated Vacuum Corp, Rochester, N.Y.
Model	HS6-1500 type 162 water-cooled cap over top jet; 3 diffusion jets; 1 ejector jet; vapor separator to bottom diffusion jet; all aluminum jets	PMC-1440A: conduction and radiantly cooled; cap over top jet; 4 aluminum diffusion jets
Date purchased	August 1962	April 1964
Oil charge	400 cc	800 cc
Heater	1500 watts, flat plate	1800 watts, 6 cartridges, thermal siphon type
Material	Stainless steel shell; aluminum jet stacks	Mild steel shell; aluminum jet stacks
Nominal speed	1500 liters/sec, air	1520 liters/sec, air
Forepump tolerance	0.75 torr	0.6 torr, no leak 0.35 torr, full load

[a]Reference 37.
[b]Reference 6.

Table 6

SPECIFICATIONS OF BAFFLES

	CVC-BC-61	Right-Angle Elbow	NRC-HN-6	G/P-251
Manufacturer	Consolidated Vacuum Corp.	IIT Research Institute	NRC Equipment Co.	Granville Phillips Co.
Type	Chevron BC 61	Length of Legs: 5% over line of sight requirement	Cryo No. 0315-6	Cryosorb 251-001-276-277
Material	Aluminum body, nickel-plated copper chevrons, stainless steel reservoir	Mild steel	Nickel-plated	Type 304 stainless steel
Conductance, liters/sec (air)	1030[a]	1000[b]	1200[c]	475[d]
Transmittance probability	0.25[e]	0.27[f]	0.30[f]	0.21[f]
Average number of collisions	7.9[f]	9.6[f]	7.4[f]	7.4[f]

[a] Reference 8.
[b] Reference 31.
[c] Reference 37.
[d] Reference 19.
[e] Reference 20.
[f] Reference 32.

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Table 7

SPECIFICATIONS OF DIFFUSION PUMP OILS

	DC 705[a]	Convalex 10[b]	OS 124[c]
Composition	1,1,3,5,5-(phenyl)₅-1,3,5-(methyl)₃-trisiloxane	Mixed pentaphenyl ethers subjected to proprietary purification 80% mmm 5-ring PPE 20% (mmp,pmp) 5-ring PPE	Mixed polyphenyl ethers 0.4% mixed 4-ring PPE 57.3% mmm 5-ring PPE 30.2% mmp 5-ring PPE 4.7% pmp 5-ring PPE 0.5% misc 5-ring PPE 0.3% 6-ring PPE
Supplier	Dow Corning Corp.	Consolidated Vacuum Corp.	Monsanto Co.
Purchase price	$225/gal	$458/gal	$165/gal
Molecular weight	546	447	447
Molecular diameter, cm	1.09×10^{-7}	0.99×10^{-7}	0.99×10^{-7}
Wt. of monomolecular layer, mg/cm²	1.00×10^{-4}	0.82×10^{-4}	0.82×10^{-4}
Density, g/ml, at 25°C	1.09	1.2	1.2
Refractive Index	1.579		1.631
Freezing point, °C	-15		4 pour point
Vapor pressure, torr			
at 5°C	8×10^{-12}	5×10^{-11}	
at 25°C	4×10^{-10}	1.7×10^{-9}	
at 200°C	4×10^{-2}	1×10^{-2}	
at 300°C	10	1.0	3.3
Heat of Vaporization kcal/gmole at 200°C	28.2		22

[a]References 11 and 12.

[b]References 22, 35, and 38.

[c]References 22, 35, 36, and 38.

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Table 8

SPECIFICATIONS OF COLLECTION PLATES

I.   Stainless steel plate: 13-1/2 x 1/16.
       Mounted to flange by machine screw. Cooling is by
       conduction through the steel flange plate with external
       water-cooled coils where noted. Area of wetted portion
       is 248 cm^2.

II.   Glass plate: 17 x 1 in.
       Area of wetted portion is 248 cm^2. Used for visual
       identification.

III.   Zinc plate: oval configuration.
       Area is 150 cm^2. 1/4-in. copper coil soft-soldered to
       back of first internally cooled plate.

IV.   Copper plate: 5-3/4 to 6 in. circle, 1/8 in. thick.
       Area is 180 cm^2. 1/4 in. copper coil soft-soldered
       to back.

V.   Stainless steel: 7 x 1/4 in.
       Area is 248 cm^2. 1/4 in. stainless steel tubing welded
       to back of plate.

VI.   Stainless steel: 6 x 3/8 in.
       Area is 147 cm^2. 1/2 in. stainless steel tubing welded
       to back of plate. Plate has 1-in. opening in center for
       gas analysis probe.

Table 9

<u>SPECIFICATIONS OF REFRIGERATOR</u>

Manufacturer: Webber Manufacturing Company, Inc.
                         Indianapolis, Indiana.

Type: Model WP-1500-125 convection fluid,
                         portable power pack system.

Specification: 208 volt system 3 phase, 60 cycle
                       1500 BTU per hour capacity at -125°F.

Temperature Range: Ambient to -125°F.

Convection Fluid: Methyl alcohol
                       temperature: -25 to -100°F.

Purchased: March 1963

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Table 10

SPECIFICATIONS OF GAUGE TUBES

Thermocouple gauge:

    Consolidated Vacuum Corp., Rochester, New York
    Type: GTC 105 thermocouple gauge
    Range: 1 to 1000 Hg
    Sensing tube: GTC-004
    Power: 15 watts

Ionization gauges:

    Consolidated Vacuum Corp., Rochester, New York

        GIC-011 tube: Bayard-Alpert type
                   dual filament; sensitivity 100 μamp per μ
                   at an emission current of 10 mamp;
                   X-ray limit 9×10^{-12} mm Hg.

        GIC-017 tube: Bayard-Alpert type;
                   dual filament; emission current 6 to
                   8 mamp; X-ray limit 1×10^{-10} mm Hg.

    Vacuum Products Division, Varian Associates, Palo Alto, California

        UHV-12P tube: Bayard-Alpert type
                   dual filament; emission current 4 mamp;
                   closed grid cage degases by ion bombardment
                   only; X-ray limit 2×10^{-11} mm Hg.

Table 11

SPECIFICATIONS OF IONIZATION GAUGE CONTROL UNIT

Manufacturer:	Vacuum Products Division, Varian Associates, Palo Alto, California
Type:	Model No. 971-0003
Specification:	Input Power 60 cycle AC, 115 volts, 250 watts
Grid Voltage:	130 volts
Collector Voltage:	-45 volts
Filament Current:	0 to 10 amp
Voltage:	0 to 12 volts
Pressure Range:	1×10^{-3} to 2×10^{-13} mm Hg; corresponds to a current sensitivity of 2×10^{-13} amp
Degas:	Ion bombardment 100 /mamp at 350 volts
Electrometer:	10^{-11} amp fs

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Table 12

SPECIFICATIONS OF MASS SPECTROMETER

1. General Electric Partial Pressure Analyzer

   a. Electronic control, fast and slow scan with
      cable and recorder mounted cabinet;
      Model 22PC110- P&L-71 Serial No. 30055

   b. Metal analyzer tube, straight tubulation
      1 in. diameter, 305 L, stainless steel
      Model 22PT110-P&L-71 Serial No. -62
      Electron multiplier numbers: Z6-8007, 6413, 183-6

   c. 3KG magnet (2.85 KG)
      Model 22PM103- P&L-71 Serial No. 4016

   d. 5KG magnet (4.8 KG)
      Model 22PM105- P&L-71 Serial No. 5056

   e. Acceleration voltmeter after 11/20/64
      Assemply Product Instrument, super
      calibrated Taut Band, 50 μamp
      catalog No. 65F302 (Allied Radio Corporation)

2. Hewlett Packard Oscilloscope

   a. Model 130C, Serial No. 425-02474

   b. Input impedance, 1 megohm shunted by 45 pf

   c. Maximum sensitivity, 0.2 mv/cm

3. Standard operating conditions (set for maximum N_2 sensitivity)

   a. Emission current, 1 mamp (tungsten, pins 1 and 2)

   b. Electron voltage, 70 volts

   c. Drawout, 4.6 units

   d. Deflection 1, 4.7 units

   e. Deflection 2, 5.0 units

   f. Electron multiplier, 2000 volts (setting No. 4)

   g. Scan rate: ammeter - 10 min; oscilloscope - 0.1 sec

Table 13

OPERATING SCHEDULE FOR TEST STATION NO. 1

Item	Dates		Run No.
Pumps			
HS6-1500 (wcc)[a]	62-63		
PMC-1440A (rcc)[b]	64-66		
Oils			
DC 704	11/62-3/63		
Convalex 10	7/64-1-66		R-26
Baffles			
BC-61	62-63		
	7/64-1/66		R-26
Elbow	7/64-8/64		R-35
BC-61	8/64-3/65		R-51
BC-61, cold sides	3/65-1/66		R-114
Test Head			
AEDC (ref. 28)	62-63		
Adapter flange	63		
Adapter flange	64-65		
Adapter flange	65		
Spool and adapter flange	65-66		
Collection Plate			
Stainless steel AEDC (ref. 28)	62-63	area	248 cm^2
Glass AEDC (ref. 28)	63	area	248 cm^2
Internally cooled zinc	63	area	150 cm^2
Internally cooled copper	64-65	area	180 cm^2
Internally cooled stainless steel	65-66	area	248 cm^2

[a] wcc = water-cooled cap.

[b] rcc = radiant-cooled cap.

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Table 14

OPERATING SCHEDULE FOR TEST STATION 2

Item	Dates	Run No.
Pumps		
HS6-1500 (wcc)[a]	62-66	
Oils		
DC 705		
Original Oil	11/62-2/65	R-14
Fresh Charge	2/65-1/66	R-104
DC 705 99%		
DC 704 1%	2/66-3/66	R-232
Baffles		
BC-61	11/62-2/65	R-14
BC-61 cold sides	2/65-3/66	R-104
Test Heads		
AEDC (ref. 28)	62-63	
Flanged plate	64	
Spool and flanged plate	64-66	
Collection Plate		
Stainless Steel AEDC (ref. 28)	62-63 area 248 cm^2	
Internally cooled Stainless	64-66 area 248 cm^2	

[a] wcc = water-cooled cap.

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Table 15

OPERATING SCHEDULE FOR TEST STATION 3

Item	Dates	Run No.
Pumps		
HS6-1500 (wcc)[a]	62-66	
Oils		
OS 124	62	
Convalex 10	62-6/65	R-15
Fresh Oil	6/65-11/65	R-164
Additional Fresh Oil	11/65-3/66	R-205
Baffles		
BC-61	62-7/64	R-15
HN-6D	7/64-11/64	R-37
New HN-6	11/64-3/65	R-85
HN-6 Cold sides	3/65-11/65	R-128
BC-61	11/65-11/65	R-205
BC-61 Cold sides	11/65-12/65	R-210
HN-6	12/65-3/66	R-216
Test Head		
AEDC (ref. 28)	62-64	
Spool and flanged plate	64-66	
Collection Plate		
Stainless Steel AEDC (ref. 28)	62-63	area 248 cm^2
Glass AEDC (ref. 28)	63	area 248 cm^2
Internally cooled copper	64	area 180 cm^2
Internally cooled stainless	64-66	area 248 cm^2

[a] wcc = water-cooled cap.

[b] side stream stripped added, total volume of oil 650 cc.

Table 16

OPERATING SCHEDULE FOR TEST STATION 4

Item	Dates	Run No.
Pumps		
HS6-1500 (wcc)[a]	62-66	
Oils		
DC 705	62-5/65	R-16
Fresh charge DC 705	5/65-3/66	R-154
Baffles		
Right Angle Elbow	62-5/65	R-16
G/P trap	5/65-9/65	R-154
New G/P trap	2/66-2/66	R-230
BC-61	2/66-3/66	R-237
Test Head		
AEDC (ref. 28)	62-65	
Spool and flanged plate	65-66	
Collection Plate		
Stainless Steel plate AEDC (ref. 28)	62-64 area 248 cm^2	
Internally cooled copper	64-65 area 180 cm^2	
Internally cooled stainless	65-66 area 248 cm^2	

[a] wcc = water-cooled cap.

Table 17

OPERATING SCHEDULE FOR TEST STATION 5

Item	Dates	Run No.
Pumps		
HS6-1500 (wcc)[a]	62-66	
Oils		
OS 124	62-63	
Convalex 10	63-6/64	
Fresh charge Convalex 10	6/64-2/65	R-20
OS 124	2/65-1/66	R-138
Baffles		
Right Angle Elbow	62-1/66	R-20
Test Head		
AEDC (ref. 28)	62-64	
Spool	64-66	
Collection Plate		
Stainless Steel plate AEDC (ref. 28)	62-64 area 248 cm^2	
Internally cooled copper	64-65 area 180 cm^2	
Internally cooled stainless	65-66 area 248 cm^2	

[a] wcc = water-cooled cap.

Table 18

STANDARD OPERATING PROCEDURE

1. Reduce system pressure to below .05 torr with forepump.

2. Cool baffle.

3. Heat collection plate to 212°F.

4. Start diffusion pump with 50% rated heat input for 1-1/2 hr.

5. Increase diffusion pump to 100% rated heat input.

6. Cool collection plate after 18 hr of heating.

7. Run time (after item 6) should be at least 70 hr.

8. Turn off heat to diffusion pump.

9. After diffusion pump has cooled turn off cooling to baffle and collection plate.

10. After baffle and collection plate have warmed to room temperature bleed nitrogen into fore line to re-pressure the system.

11. Remove collection plate and wash off oil with solvent. Collect one sample of backstreamed oil and two samples of analytical blanks.

12. Close system as soon as possible to prevent moisture adsorption on walls of system.

Table 19

CALIBRATION DATA FOR ULTRAVIOLET ABSORBANCE

Oil	Absorbance Band, m	Concentration, mg/ml	Absorbance Units	Slope	Deviation, %
DC 705 and DC 704	258	0.0405	0.104	0.389	0.5
		0.101	0.258	0.391	0.0
		0.202	0.521	0.388	0.8
		0.304	0.768	0.396	1.3
				avg 0.391	0.6
Convalex 10 and OS 124	278	0.0412	0.662	0.0625	0.6
		0.0206	0.328	0.0628	0.2
		0.0103	0.163	0.0632	0.5
		0.0248	0.391	0.0634	0.8
				avg 0.0629	0.5

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Table 20

ACCURACY OF ULTRA VIOLET ANALYTICAL METHOD

Oil	Weight by Seeding, mg	mg/cm^2	Rinse Volume, ml	Absorbance	Total, mg/cm^2	Analytical Blank, mg/cm^2	Net Weight, mg/cm^2	% Error
Convalex 10	1.032	57.5×10^{-4} [a]	50	0.325	57.6×10^{-4}		57.6×10^{-4} [c]	0.17
	0.775	43.2×10^{-4} [a]	25	0.475	41.7×10^{-4}		41.7×10^{-4} [c]	-3.47
	0.310	17.3×10^{-4} [a]	25	0.190	16.7×10^{-4}		16.7×10^{-4} [c]	-3.47
	0.103	5.7×10^{-4} [a]	25	0.075	6.5×10^{-4}		6.5×10^{-4} [c]	14.0
	0.051	3.49×10^{-4} [b]	10	0.090	3.88×10^{-4}	0.27×10^{-4}	3.61×10^{-4}	3.44
	0.034	2.31×10^{-4} [b]	10	0.052	2.25×10^{-4}	0.00×10^{-4}	2.25×10^{-4}	-2.59
	0.034	2.31×10^{-4} [b]	10	0.056	2.38×10^{-4}	0.00×10^{-4}	2.38×10^{-4}	3.02
	0.0104	0.71×10^{-4} [b]	10	0.027	1.15×10^{-4}	0.51×10^{-4}	0.064×10^{-4}	-9.84
								avg 4.5
DC 705	1.013	56.9×10^{-4} [a]	25	0.109	56.5×10^{-4}		56.5×10^{-4} [c]	0.18
	0.912	50.6×10^{-4} [a]	25	0.099	53.6×10^{-4}		53.6×10^{-4} [c]	5.93
	0.678	37.6×10^{-4} [a]	25	0.06	32.6×10^{-4}		32.6×10^{-4} [c]	-13.28
	0.304	16.9×10^{-4} [a]	25	0.04	21.6×10^{-4}		21.6×10^{-4} [c]	27.75
	0.055	3.75×10^{-4} [b]	10	0.026	6.90×10^{-4}	2.66×10^{-4}	4.24×10^{-4}	13.05
	0.055	3.75×10^{-4} [b]	10	0.020	5.31×10^{-4}	1.86×10^{-4}	3.45×10^{-4}	-8.00
	0.033	2.31×10^{-4} [b]	10	0.016	4.25×10^{-4}	0.00×10^{-4}	4.25×10^{-4}	84.0
	0.022	1.50×10^{-4} [b]	10	0.011	2.66×10^{-4}	1.33×10^{-4}	1.33×10^{-4}	-11.34
								avg 29.0

[a] Area of sampling plate 180 cm^2.
[b] Area of sampling plate 147 cm^2.
[c] Uncorrected values.

Table 21

RECOVERY WEIGHT OF POLYPHENYL ETHERS BY ULTRAVIOLET, GAS CHROMATOGRAPHIC
AND GRAVIMETRIC TECHNIQUES

Station	Run No.	Deposit, mg/cm²		Rate, mg/cm²-min		% Recovery	% Recovery[a] by nonpolar Chromotography
		Ultraviolet	Weight	Ultraviolet	Weight	UV/Wt.	
1	55	387×10^{-4}	635×10^{-4}	7×10^{-6}	11.5×10^{-6}	60.8	40
3	57	1513×10^{-4}	1578×10^{-4}	28×10^{-6}	29×10^{-6}	96.6	90
5	58	341×10^{-4}	437×10^{-4}	6×10^{-6}	8×10^{-6}	75.1	78

[a]Determined from Figure 23

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Table 22

RECOVERY OF STANDARDS USED FOR GAS CHROMATOGRAPHY CALIBRATION

% DC 704 in DC 705	DC 704		DC 705	
	Area, cm^2	% Recovered	Area, cm^2	% Recovered
50	9.75 avg	100	9.42 avg	100
	3.27	33.6	3.30	35.0
	3.42	35.1	3.27	34.7
	4.10	42.1	4.20	44.6
	4.35	44.6	4.45	47.2
		avg 38.9		avg 40.4
20	2.40 avg	100	10.02 avg	100
	2.1	88.5	8.6	85.8
	2.72	113.8	10.9	108.5
	2.30	96.9	9.2	91.8
		avg 99.7		avg 95.4
9.1	0.81 avg	100	8.08 avg	100
	0.58	69.7	5.70	70.6
	0.55	66.7	5.85	72.8
	0.60	72.7	6.18	76.5
		avg 69.7		avg 73.3
2.4	0.92 avg	100	8.68 avg	100
	1.06	114.3	7.05	83.2
	1.01	109.2	7.40	87.2
	0.90	97.7	7.02	82.7
		avg 107.1		avg 84.6

Overall average of recovery was 76%.

Table 23

SUMMARY OF SENSITIVITIES FOR MASS SPECTROMETER

Gas	Mass (M)	Sensitivities				Sensitivity[a] of B-A Gauge, Relative/N_2
		Small Magnet		Large Magnet		
		Amp/torr	Relative/N_2	Amp/torr	Relative/N_2	
Hydrogen	2	0.4	6.7			0.5
Helium	4	0.015	0.25			0.16
Water	18	0.18	3.0	0.25	2.8	1.1
Nitrogen	28	0.06	1.0	0.09	1.0	1.0
Oxygen	32	0.04	0.67	0.08	0.9	0.8
MeOH	30	0.36	6.0			
Benzene	78	0.00001	0.0002	0.003	0.03	

[a]Reference 41.

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Table 24

PERFORMANCE UNDER STANDARD CONDITIONS
(Station 1, PMC-1440A Pump, Convalex 10 Oil)

Run	Length of Run, hr	Baffle	System Pressure, torr		Deposit Weight, mg/cm²		Backstreaming Values	
			at Room Temp.	at LN_2 Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
139	0	BC-61	9×10^{-8}		0.7×10^{-4}			
151	0	BC-61	2×10^{-7}		0.41×10^{-4}	0.38×10^{-4}	0.03×10^{-4}	
117	74	BC-61	8×10^{-8}	6×10^{-9}	1.06×10^{-4}			0.024×10^{-6}
121	74	BC-61	7×10^{-8}	9×10^{-9}	1.06×10^{-4}			0.024×10^{-6}
160	76	BC-61	2×10^{-7}	9×10^{-9}	0.80×10^{-4}	0.26×10^{-4}	0.54×10^{-4}	0.012×10^{-6}
180	75	BC-61	1×10^{-7}	1×10^{-9}	1.02×10^{-4}	0	1.02×10^{-4}	0.023×10^{-6}
148	142	BC-61	2×10^{-7}	4×10^{-9}	2.74×10^{-4}			0.032×10^{-6}
156	295	BC-61	2×10^{-7}	1×10^{-9}	0.80×10^{-4}	0.4×10^{-4}	0.40×10^{-4}	0.003×10^{-6}
174	292	BC-61	1×10^{-7}	1×10^{-9}	1.27×10^{-4}	0.25×10^{-4}	1.02×10^{-4}	0.006×10^{-6}
184	292	BC-61	4×10^{-7}	9×10^{-10}	1.04×10^{-4}	0.25×10^{-4}	0.79×10^{-4}	0.005×10^{-6}
202	306	BC-61	5×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.13×10^{-4}	0.89×10^{-4}	0.005×10^{-6}
218	486	BC-61	1×10^{-7}	1×10^{-9}	1.02×10^{-4}	0	1.02×10^{-4}	0.004×10^{-6}
208	512	BC-61	3×10^{-7}	5×10^{-10}	1.27×10^{-4}	0	1.27×10^{-4}	0.004×10^{-6}

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Table 25

PERFORMANCE UNDER STANDARD CONDITIONS
(Station 2, HS6-1500 Pump, DC 705 Oil)

Run	Length of Run, hr	Baffle	System Pressure, torr		Deposit Weight, mg/cm^2		Backstreaming Values	
			at Room Temp.	at LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm^2	Rate, mg/cm^2-min
137	0	BC-61			3.2×10^{-4}			
142	0	BC-61	4×10^{-8}		2.5×10^{-4}			
143	0	BC-61	6×10^{-8}		1.9×10^{-4}			
118	74	BC-61	6×10^{-9}	1×10^{-9}	1.9×10^{-4}			0.043×10^{-6}
135	74	BC-61	3×10^{-9}	1×10^{-9}	3.2×10^{-4}			0.071×10^{-6}
161	74	BC-61	9×10^{-9}	1×10^{-9}	2.5×10^{-4}	0.8×10^{-4}	1.7×10^{-4}	0.039×10^{-6}
181	75	BC-61	7×10^{-9}	1.57×10^{-4}	1.57×10^{-4}	1.57×10^{-4}	0	0
149	145	BC-61	4×10^{-9}	9×10^{-10}	3.2×10^{-4}	2.2×10^{-4}	1.0×10^{-4}	0.011×10^{-6}
225	126	BC-61	7×10^{-9}	1×10^{-9}	3.17×10^{-4}	0.79×10^{-4}	2.38×10^{-4}	0.027×10^{-6}
157	293	BC-61	5×10^{-9}	5×10^{-10}	1.9×10^{-4}	0.9×10^{-4}	1.0×10^{-4}	0.005×10^{-6}
172	292	BC-61	5×10^{-9}	8×10^{-10}	3.2×10^{-4}	1.57×10^{-4}	1.63×10^{-4}	0.009×10^{-6}
185	291	BC-61	4×10^{-9}	7×10^{-10}	4.73×10^{-4}	3.17×10^{-4}	1.56×10^{-4}	0.009×10^{-6}
203	306	BC-61	3×10^{-9}	6×10^{-10}	7.62×10^{-4}	1.57×10^{-4}	6.05×10^{-4}	0.033×10^{-6}
219	486	BC-61	3×10^{-9}	7×10^{-10}	4.75×10^{-4}	2.40×10^{-4}	2.35×10^{-4}	0.008×10^{-6}
207	512	BC-61	3×10^{-9}	7×10^{-10}	3.17×10^{-4}	0	3.17×10^{-4}	0.010×10^{-6}

Table 26

PERFORMANCE UNDER STANDARD CONDITIONS

(Station 3, HS6-1500 Pump, Convalex 10 Oil)

Run	Length of Run, hr	Baffle	System Pressure, torr		Deposit Weight, mg/cm^2		Backstreaming Values	
			at Room Temp.	at LN_2 Temp.	Total	Analytical Blank	Deposit, mg/cm^2	Rate, mg/cm^2-min
238	67	HN-6	8×10^{-9}	2×10^{-9}	7.50×10^{-4}	0.28×10^{-4}	7.22×10^{-4}	0.180×10^{-6}
235	77	HN-6	8×10^{-9}	2×10^{-9}	7.50×10^{-4}	0.13×10^{-4}	7.37×10^{-4}	0.158×10^{-6}
223	146	HN-6	5×10^{-9}	2×10^{-9}	7.41×10^{-4}	0.25×10^{-4}	7.16×10^{-4}	0.082×10^{-6}
233	150	HN-6	4×10^{-9}	2×10^{-9}	9.50×10^{-4}	0.23×10^{-4}	9.27×10^{-4}	0.102×10^{-6}
210	148	BC-61	2×10^{-8}	2×10^{-9}	0.92×10^{-4}	0	0.92×10^{-4}	0.010×10^{-6}
228	250	HN-6	5×10^{-9}	2×10^{-9}	13.70×10^{-4}	0.15×10^{-4}	13.55×10^{-4}	0.090×10^{-5}
216	293	HN-6	3×10^{-9}	2×10^{-9}	12.00×10^{-4}	0.15×10^{-4}	11.85×10^{-4}	0.067×10^{-5}

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Table 27

PERFORMANCE UNDER STANDARD CONDITIONS

(Station 4, HS6-1500 Pump, DC 705 Oil)

Run	Length of Run, hr	Baffle	System Pressure, torr		Deposit Weight, mg/cm²		Backstreaming Values	
			at Room Temp.	at LN_2 Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
127	0	RAE	4×10^{-8}		15.6×10^{-4}			
132	0	RAE			14.1×10^{-4}			
119	74	RAE	7×10^{-9}	2×10^{-9}	21.8×10^{-4}			0.493×10^{-6}
123	74	RAE	4×10^{-9}	3×10^{-9}	23.9×10^{-4}			0.542×10^{-6}
134	74	RAE	5×10^{-9}	1×10^{-9}	18.3×10^{-4}	2.17×10^{-4}	16.13×10^{-4}	0.363×10^{-6}
140	76	RAE	4×10^{-9}	9×10^{-10}	23.5×10^{-4}			0.520×10^{-6}
159	51	G/P	2×10^{-8}	6×10^{-9}	21.5×10^{-4}	10.8×10^{-4}	10.7×10^{-4}	0.349×10^{-6}
155	56	G/P	9×10^{-9}	6×10^{-9}	17.8×10^{-4}	4.3×10^{-4}	13.5×10^{-4}	0.408×10^{-6}
231	80	G/P	4×10^{-8}	3×10^{-9}	11.2×10^{-4}	2.8×10^{-4}	8.4×10^{-4}	0.174×10^{-6}
237	67	BC-61	2×10^{-7}	2×10^{-9}	3.48×10^{-4}	0.79×10^{-4}	2.69×10^{-4}	0.066×10^{-6}
240	71	BC-61	3×10^{-7}	1×10^{-9}	3.31×10^{-4}	1.26×10^{-4}	2.05×10^{-4}	0.048×10^{-6}
236	126	G/P	4×10^{-8}	4×10^{-9}	16.1×10^{-4}	3.6×10^{-4}	12.5×10^{-4}	0.166×10^{-6}
230	150	G/P	5×10^{-8}	9×10^{-9}	24.6×10^{-4}	6.5×10^{-4}	18.1×10^{-4}	0.199×10^{-6}
244	167	BC-61	7×10^{-7}	2×10^{-9}	3.49×10^{-4}	0	3.49×10^{-4}	0.035×10^{-6}

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Table 28

PERFORMANCE UNDER STANDARD CONDITIONS
(Station 5, HS6-1500 Pump, OS 124 Oil)

Run	Length of Run, Hr	Baffle	System Pressure, torr		Deposit Weight, mg/cm²		Backstreaming Values	
			at Room Temp.	at LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
141	75	RAE	1×10^{-8}	5×10^{-9}	0.51×10^{-4}			0.015×10^{-6}
165	75	RAE	1×10^{-8}	4×10^{-9}	0.71×10^{-4}	0.36×10^{-4}	0.35×10^{-4}	0.006×10^{-6}
150	145	RAE	8×10^{-8}	8×10^{-8}	1.12×10^{-4}	0.51×10^{-4}	0.61×10^{-4}	0.007×10^{-6}
183	148	RAE	5×10^{-9}	4×10^{-9}	0.92×10^{-4}	0.25×10^{-4}	0.67×10^{-4}	0.008×10^{-6}
170	290	RAE	4×10^{-9}	2×10^{-9}	1.10×10^{-4}	0.10×10^{-4}	1.00×10^{-4}	0.006×10^{-6}
193	292	RAE	6×10^{-8}	5×10^{-8}	1.36×10^{-4}	0.46×10^{-4}	0.90×10^{-4}	0.005×10^{-6}
188	294	RAE	3×10^{-9}	1×10^{-9}	1.30×10^{-4}	0.25×10^{-4}	1.05×10^{-4}	0.006×10^{-6}

Table 29

CATALYTIC DECOMPOSITION EFFECTS AT STATION 3

Run	Oil Type	Oil Age	Length of Run, hr	Baffle[a]	Weight of Oil, mg/cm	Backstreaming Rate, mg/cm²-min	Conditions
128	Convalex 10	2 yr	74	HN-6D*	6.3×10^{-4} uc	0.14×10^{-6}	SC
133	Convalex 10	2 yr	74	HN-6D*	6.8×10^{-4} uc	0.15×10^{-6}	SC
	Cleaned system with solvent, new oil, new HN-6 baffle						
164	Convalex 10	75 hr	75	HN-6	10.2×10^{-4} c	0.23×10^{-6}	LE
169	Convalex 10	370 hr	290	HN-6	21.9×10^{-4} c	0.13×10^{-6}	SC
177	Convalex 10	900 hr	313	HN-6	40.9×10^{-4} c	0.22×10^{-6}	SC
186	Convalex 10	1500 hr	292	HN-6	49.0×10^{-4} c	0.28×10^{-6}	SC
	Changed jet stack, new oil, BC-61 baffle						
205	Convalex 10	411 hr	411	BC-61	5.2×10^{-4} c	0.02×10^{-6}	Warm sides
210	Convalex 10	550 hr	148	BC-61	0.9×10^{-4} c	0.01×10^{-6}	SC
	Replaced HN-6 baffle						
216	Convalex 10	900 hr	293	HN-6	11.9×10^{-4} c	0.07×10^{-6}	SC
223	Convalex 10	1100 hr	146	HN-6	7.2×10^{-4} c	0.08×10^{-6}	SC
238	Convalex 10	>2000 hr	67	HN-6	7.2×10^{-4} c	0.18×10^{-6}	SC

[a]HN-6D* is a defective baffle.

Note: uc = uncorrected for analytical blank; c = corrected for analytical blank;
SC = Standard Condition run; and LE = Light end run.

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Table 30

CATALYTIC DECOMPOSITION EFFECTS AT STATION 4

Run	Oil Type	Oil Age	Length of Run, hr	Baffle[a]	Weight of Oil, mg/cm^2		Backstreaming Rate, mg/cm^2-min	Conditions
34	DC 705	2 yr	360	RAE (-75°F)	182 $\times 10^{-4}$	uc	0.84 $\times 10^{-6}$	Not SC
119	DC 705	2 yr	74	RAE (-75°F)	21.8 $\times 10^{-4}$	uc	0.49 $\times 10^{-6}$	SC
123	DC 705	2 yr	74	RAE (-75°F)	23.9 $\times 10^{-4}$	uc	0.54 $\times 10^{-6}$	SC
134	DC 705	2 yr	74	RAE (-75°F)	16.1 $\times 10^{-4}$	c	0.36 $\times 10^{-6}$	SC
				New Oil Added G/P Cryosorb baffle				
155	DC 705	173 hr	56	G/P LN$_2$	13.5 $\times 10^{-4}$	c	0.41 $\times 10^{-6}$	Not SC
159	DC 705	224 hr	51	G/P LN$_2$	10.7 $\times 10^{-4}$	c	0.35 $\times 10^{-6}$	Not SC
163	DC 705	319 hr	95	G/P LN$_2$	15.9 $\times 10^{-4}$	c	0.29 $\times 10^{-6}$	Not SC
236	DC 705	1405 hr	126	G/P (-75°F)	12.5 $\times 10^{-4}$	c	0.17 $\times 10^{-6}$	SC
				Added BC-61 Baffle				
237	DC 705	1472 hr	67	BC-61	2.7 $\times 10^{-4}$	c	0.07 $\times 10^{-6}$	SC
240	DC 705	1543 hr	71	BC-61	2.1 $\times 10^{-4}$	c	0.05 $\times 10^{-6}$	SC
244	DC 705	1710 hr	167	BC-61	3.5 $\times 10^{-4}$	c	0.04 $\times 10^{-6}$	SC

[a]HN-6D* is a defective baffle

Note: uc = uncorrected for analytical blank; c = corrected for analytical blank;
      SC = Standard Condition run; and LE = Light end run.

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Table 31

RELATIVE DISTRIBUTION OF MAJOR ISOMERIC COMPONENTS
IN UNUSED POLYPHENYL ETHER FLUIDS

Sample	Area, %[b]				
	A mmm	B	C	D pmp	E mmp
OS 124-1	57.1	37.1	5.8		
OS 124-2	61.0	35.0	4.0		
OS 124-3	66.5	29.2	4.3		
OS 124-4	65.2	31.1	3.4		
OS 124-5	62.2	33.7	4.9		
Convalex 10-1	87.3			5.5	7.2
Convalex 10-2[c]	77.5			6.9	15.4

[a]Both OS-124 and Convalex-10 fluids contained a light component equivalent to approximately 0.2% of the total area.

[b]The distribution of components is based on the area of each peak as measured by a compensating polar planimeter.

[c]Fresh sample used in backstreaming studies.

Table 32

ISOMER DISTRIBUTION FOR CONVALEX 10[a]
FROM STATION NO. 3 AFTER RUN 133 IN USE FOR
2 YEARS

Isomer	%	
	Monsanto (ref. 35)	IITRI (ref. 38)
4-ring PPE (mm)	0.3	
4-ring PPE (mg)	0.1	
5-ring PPE (mmm)	57.3	77-87
5-ring PPE (mmp)	30.3	7-15
5-ring PPE (pmp)	4.7	5-7
5-ring PPE (misc)	0.5	
6-ring PPE	0.3	
Residue	6.6	

[a]Analyzed sample contained 0.015 mole percent phenols, which is quite acceptable for a used polyphenyl ether. Normal production of OS 124 was somewhat lower.

Table 33

EFFECT OF ADDED LIGHT END DC704 TO DC705 IN BACKSTREAMING MEASUREMENTS

Run	Station	Oil	Bakeout Temp., °F	Length of Run	Trap Temp., °F	System Pressure, torr		Deposit Weight mg/cm²		Net Backstreaming	
						Room Temp.	LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
181	2	DC 705	212	75	-76	7×10^{-9}	1×10^{-9}	1.57×10^{-4}	1.57×10^{-4}	0	0
203	2	DC 705	212	306	-76	3×10^{-9}	6×10^{-10}	7.60×10^{-4}	1.57×10^{-4}	6.03×10^{-4}	0.0327×10^{-6}
207	2	DC 705	212	512	-60	3×10^{-9}	7×10^{-10}	3.17×10^{-4}	0	3.17×10^{-4}	0.0103×10^{-6}
219	2	DC 705	212	486	-75	3×10^{-9}	7×10^{-10}	4.75×10^{-4}	2.40×10^{-4}	2.35×10^{-4}	0.0081×10^{-6}
225	2	DC 705	212	126	-75	5×10^{-9}	1×10^{-9}	2.85×10^{-4}	0	2.85×10^{-4}	0.038×10^{-6}
232	2	DC 704/DC 705[a]	212	150	-75	5×10^{-9}	1×10^{-9}	2.40×10^{-4}	0.32×10^{-4}	2.08×10^{-4}	0.0228×10^{-6}
234	2	DC 704/DC 705[a]	212	77	RT[b]	1×10^{-8}	2×10^{-9}	51.7×10^{-4}	2.2×10^{-4}	49.5×10^{-4}	1.070×10^{-6}
239	2	DC 704/DC 705[a]	212	67	-75	6×10^{-9}	1×10^{-9}	1.89×10^{-4}	0.79×10^{-4}	1.10×10^{-4}	0.0275×10^{-6}
245	2	DC 704/DC 705[a]	212	295[c]	-75	9×10^{-9}	3×10^{-9}	0.79×10^{-4}	0	0.79×10^{-4}	0.0045×10^{-6}

[a]The side-stream stripper was added to the system. A mixture of 99% DC 705 and 1% DC 704 was used as a fresh charge.
[b]A light end removal run was performed.
[c]The side-stream stripper ran for 123 of the 295 hr of the run.

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Table 34

ULTRAVIOLET ANALYSIS OF MIXED DC 704/DC 705 BACKSTREAMING SAMPLES

Sample No.	Concentrated?	Wt., mg/cc	Area, cm^2 DC 704	DC 705	Total	Ratio of DC 704 to TA	% DC 704
232	no	0.00587	0.85	4.25	5.10	0.167	10.2
			2.18	8.80	10.98	0.198	10.8
					Avg	0.182	10.5
234	no	0.128	1.00	4.42	5.42	0.184	10.55
			0.98	4.20	5.18	0.188	10.65
					Avg	0.186	10.6
234	yes		0.45	1.80	2.25	0.200	10.9
			1.60	7.95	9.55	0.201	10.9
239	yes	0.0047	0.245	0.238	3.71	0.066	6.7
			0.238	3.315	3.55	0.068	6.9
					Avg	0.067	6.8

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Table 35

GAS-CHROMATOGRAPHIC TREATMENT OF MIXED DC 704/DC 705 BACKSTREAMING SAMPLES

| Sample No.	Concentrated	Sensitivity	Peak Height, cm	Wt., μg	Scale Factor	Dilution Factor	DC 705 Wt., mg	% DC 705	Total Wt., mg	Deposit, 10^{-4} mg/cm^2 GC	Deposit, 10^{-4} mg/cm^2 UV	% Difference
232	yes	8	11.35	4.70	1	10	0.047	89.5	0.0525	2.1	2.4	-14
234	no	4	5.30	2.60	1/2	1000	1.30	89.4	1.45	58.5	51.7	11.6
234	no	4	4.65	2.30	1/2	1000	1.15	89.4	1.28	52.0	51.7	0.6
				Avg 2.45	1/2	1000	1.22	89.4	1.36	55.2	51.7	6.3
234	yes	16[a]	2.2	1.10	2	1000	2.20	89.1			51.7	
234	yes	16[a]	8.35	3.65	2	200	1.46	89.1	2		51.7	
				Avg			1.83	89.1	2.05	83.6	51.7	38.1
239[b]	yes	8	3.65	1.63	1	2	0.003^6					
239[b]	yes	8	3.40	1.60	1	2	0.003^2					
				Avg 1.61	1	2	0.003^4	93.3	0.003^6	0.15	1.9	-(large)

[a] Sensitivity not recorded; 16 assumed from amount of base line noise present.

[b] Very strange sample with light ends present.

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Table 36

EFFECT OF SIDE-STREAM STRIPPER

Sample No.	History[a]	Area, cm DC 704	DC 705	Total	Ratio of DC 704 to Total	% DC 704
SSS-1	Top plate	0.36	17.94	18.30	0.0197	1.95
SSS-2	First alembic	0.32	16.29	16.61	0.0193	1.90
SSS-3	Second alembic	0.32	17.94	18.26	0.0175	1.73
SSS-4	Third alembic	0.25	14.24	14.49	0.0169	1.67
SSS-5	Return	0.35	22.24	22.59	0.0154	1.52
SSS-6	Bottom	0.24	18.52	18.76	0.0128	1.26

[a]See Figure below:
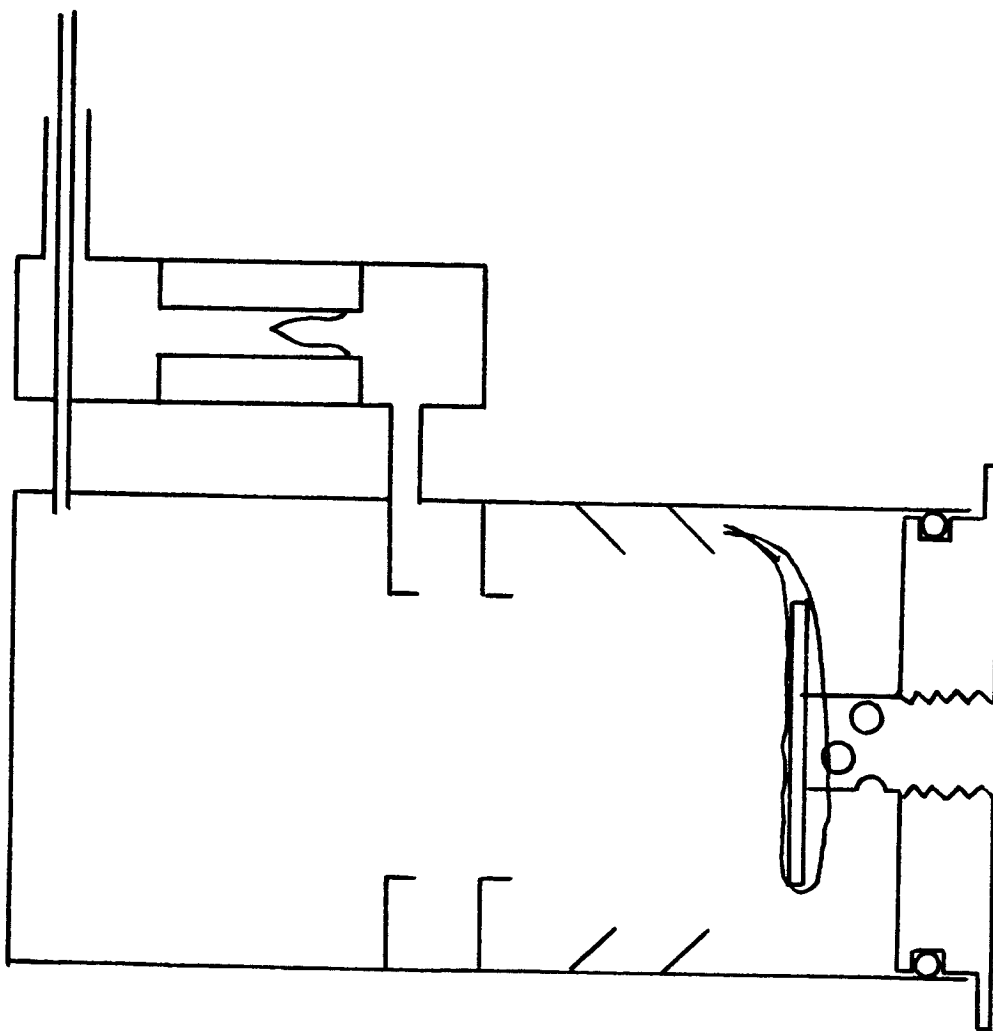
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Table 37

EVALUATION OF HN-6 BAFFLE: BACKSTREAMING MEASUREMENTS AT STATION 3

Run	Baffle	Oil	Bakeout Temp., °F	Run, hr	Trap Temp., °F	System Pressure, torr		Deposit Weight, mg/cm²		Net Backstreaming	
						at Room Temp.	at LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
216	HN-6	Convalex 10	212	293	-75	3×10^{-9}	2×10^{-9}	12.0	0.15×10^{-4}	11.85×10^{-4}	0.067×10^{-6}
223	HN-6	Convalex 10	212	146	-75	5×10^{-9}	2×10^{-9}	7.41	0.25×10^{-4}	7.16×10^{-4}	0.0815×10^{-6}
228	HN-6	Convalex 10	212	249.5	-25[a]	5×10^{-9}	2×10^{-9}	13.7	0.15×10^{-4}	13.5×10^{-4}	0.090×10^{-6}
233	HN-6	Convalex 10	212	150	-75	4×10^{-9}	2×10^{-9}	9.5	0.23×10^{-4}	9.3×10^{-4}	0.102×10^{-6}
235	HN-6	Convalex 10	212	77	-75	8×10^{-9}	2×10^{-9}	7.5	0.13×10^{-4}	7.4×10^{-4}	0.158×10^{-6}
238	HN-6	Convalex 10	212	67	-75	8×10^{-9}	2×10^{-9}	7.5	0.28×10^{-4}	7.2×10^{-4}	0.180×10^{-6}
243	HN-6	Convalex 10	212	167	-75[b]	9×10^{-9}	4×10^{-9}	9.9[b]	0.26×10^{-4}	9.6×10^{-4}[b]	0.096×10^{-6}[b]
205	BC-61	Convalex 10	212	411	-44[c]	2×10^{-8}	3×10^{-9}	5.4	0.17×10^{-4}	5.2×10^{-4}	0.0211×10^{-6}
210	BC-61	Convalex 10	212	148	-68/-10[d]	2×10^{-8}	2×10^{-9}	0.92	0.0×10^{-4}	0.92×10^{-4}	0.0103×10^{-6}

[a]Using HN-6 baffle at -25°F.

[b]Using HN-6 baffle with warm sides, trap itself at -75°F.

[c]Chevron baffle with warm sides. Refrigeration system was not cooling properly.

[d]Chevron baffle with cold sides. Refrigeration system was still not cooling properly

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Table 38

EVALUATION OF CRYOSORB BAFFLE: BACKSTREAMING MEASUREMENTS AT STATION 4

Run	Baffle	Oil	Bakeout Temp., °F	Run, hr	Trap Temp., °F	System Pressure, torr at Room Temp.	System Pressure, torr at LN$_2$ Temp.	Deposit Weight, mg/cm^2 Total	Deposit Weight, mg/cm^2 Analytical Blank	Net Backstreaming Deposit, mg/cm^2	Net Backstreaming Rate, mg/cm^2-min
231	BC-61	DC 705	212	79.5	-75	4×10^{-8}	3×10^{-9}	11.2×10^{-4}	2.8×10^{-4}	8.4×10^{-4}	0.174×10^{-6}
236	BC-61	DC 705	212	126	-75	4×10^{-8}	4×10^{-9}	16.1×10^{-4}	3.6×10^{-4}	12.5×10^{-4}	0.166×10^{-6}
237	BC-61	DC 705	212	67	-75	2×10^{-7}	2×10^{-9}	3.48×10^{-4}	0.79×10^{-4}	2.69×10^{-4}	0.066×10^{-6}
240	BC-61	DC 705	212	71	-75	3×10^{-7}	1×10^{-9}	3.31×10^{-4}	1.26×10^{-4}	2.05×10^{-4}	0.048×10^{-6}
244	BC-61	DC 705	212	167	-75	7×10^{-7}	2×10^{-9}	3.49×10^{-4}	0	3.49×10^{-4}	0.035×10^{-6}

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Table 39

OIL MIGRATION UNDER OPTIMUM CONDITIONS

Station	Baffle	Oil	Sides	Run	Length of Run, hr	Deposit, mg/cm^2	Backstreaming Rate, mg/cm^2-min
5	RAE	OS 124	Warm	147	74	5.2×10^{-4}	0.12×10^{-6}
			Cold	141	75	0.5×10^{-4}	0.02×10^{-6}
3	HN-6	Convalex 10	Warm	243	167	9.6×10^{-4}	0.10×10^{-6}
			Cold	233	77	7.4×10^{-4}	0.16×10^{-6}
3	BC-61	Convalex 10	Warm	205	411	5.4×10^{-4}	0.02×10^{-6}
			Cold	210	148	0.9×10^{-4}	0.01×10^{-6}
1	BC-61	Convalex 10	Warm	109	168	1.9×10^{-4}	0.02×10^{-6}
			Cold	117	74	1.1×10^{-4}	0.02×10^{-6}
2	BC-61	DC 705	Warm	98[a]	74	19.2×10^{-4}	0.48×10^{-6}
			Cold	108[a]	264	6.5×10^{-4}	0.04×10^{-6}

[a]No bakeout of collection plate on start up.

Table 40

DECREASE IN BACKSTREAMING WITH CONDITIONING PERIOD

Station	Oil	Baffle	Heating Time, hr	Run Time, hr	Temperature Baffle	Temperature Collection Plate	Deposit Weight mg/cm²	Backstreaming, mg/cm²-min
5	OS 124	RAE	192.5	192.5	RT	RT	46.1×10^{-4}	0.4×10^{-6}
5	OS 124	RAE	600	211	RT	RT	25.4×10^{-4}	0.2×10^{-6}
5	OS 124	RAE	960	360	RT	RT	4.3×10^{-4}	0.02×10^{-6}
5	Convalex 10	RAE	408	408	RT	RT	9.8×10^{-4}	0.04×10^{-6}
5	Convalex 10	RAE	813.5	405.5	5	5	7.3×10^{-4}	0.03×10^{-6}
5	Convalex 10	RAE	2856	1392	15	15	2.5×10^{-4}	0.003×10^{-6}
5	OS 124	RAE	1440	1440	RT	RT	3.83×10^{-4}	0.004×10^{-6}
4	DC 705	RAE	744	168	RT	RT	3030×10^{-4}	30.0×10^{-6}
4	DC 705	RAE	912	165.5	RT	RT	29.8×10^{-4}	0.3×10^{-6}
4	DC 705	RAE	1166	254.5	RT	RT	15.3×10^{-4}	0.1×10^{-6}
4	DC 705	RAE	2760	405.5	5	5	14.6×10^{-4}	0.06×10^{-6}
4	DC 705	RAE	3288	492	5	5	29.5×10^{-4}	0.1×10^{-6}
3	Convalex 10	BC-61	411	411	-44[a]	-44	5.23×10^{-4}	0.021×10^{-6}
3	Convalex 10	BC-61	550	148	-23[b]	-23	0.92×10^{-4}	0.010×10^{-6}
2	DC 705	BC-61	8760	292.5	-74[b]	-74	0.96×10^{-4}	0.005×10^{-6}
2	DC 705/704	BC-61	294	67	-75[b]	-75	1.10×10^{-4}	0.028×10^{-6}
2	DC 705/704	BC-61	589	295	-76[b]	-76	0.79×10^{-4}	0.005×10^{-6}

[a] Warm sides on baffle.
[b] Cold sides on the baffle.

Table 41

INITIAL OIL DEPOSITION DUE TO START-UP CONDITIONS

Run	Station	Oil	Bakeout Temp., °F	Heat Load to Diffusion Pump, %	Length of Run, hr	Temp. of Trap/Wall	System Pressure, torr at Room Temp.	System Pressure at LN$_2$ Temp.	Deposit Weight, mg/cm^2 Total	Analytical Blank	Net Backstreaming Deposit, mg/cm^2	Rate, mg/cm^2-min
100	1	Convalex 10	None	100	70	-76/RT	1×10^{-7}	4×10^{-9}	4.0×10^{-4}			0.10×10^{-6}
103	1	Convalex 10	None	50	74	-76/RT	5×10^{-8}	6×10^{-9}	2.4×10^{-4}			0.04×10^{-6}
113	1	Convalex 10	None	50	170	-78/RT	6×10^{-8}	6×10^{-9}	4.2×10^{-4}			0.04×10^{-6}
117	1	Convalex 10	212°	50	74	-78/14	8×10^{-8}	6×10^{-9}	1.06×10^{-4}			0.024×10^{-6}
152	1	Convalex 10	None	50	72	-77/14	3×10^{-7}	6×10^{-9}	1.40×10^{-4}	0.35×10^{-4}	1.05×10^{-4}	0.024×10^{-6}
98	2	DC 705	None	100	74	-76/RT	5×10^{-8}	3×10^{-8}	19.2×10^{-4}			0.477×10^{-6}
101	2	DC 705	None	50	71	-76/RT	5×10^{-8}	3×10^{-8}	18.3×10^{-4}			0.427×10^{-6}
107	2	DC 705	None	50	151	-77/14	6×10^{-9}	2×10^{-8}	5.0×10^{-4}			0.056×10^{-6}
108[a]	2	DC 705	None	50	264	-75/14	7×10^{-9}	2×10^{-9}	6.5×10^{-4}			0.041×10^{-6}
112	2	DC 705	None	50 (X3)	146	-78/14	6×10^{-9}	1×10^{-9}	4.0×10^{-4}			0.045×10^{-6}
118[b]	2	DC 705	212	50	74	-75/14	8×10^{-9}	2×10^{-9}	1.9×10^{-4}			0.043×10^{-6}
125[b]	2	DC 705	212	100	74	-75/14	8×10^{-9}	2×10^{-9}	4.0×10^{-4}			0.088×10^{-6}
153	2	DC 705	None	50	146	-77/14	4×10^{-9}	7×10^{-10}	3.2×10^{-4}	1.6×10^{-4}	1.6×10^{-4}	0.018×10^{-6}
88	5	Convalex 10	None	100	141	-75	7×10^{-8}	6×10^{-9}	4.1×10^{-4}			0.05×10^{-6}
89[c]	5	Convalex 10	None	100	128	-75	1×10^{-8}	4×10^{-9}	3.9×10^{-4}			0.05×10^{-6}
92[c]	5	Convalex 10	None	100 (X2)	69	-76	2×10^{-8}	7×10^{-9}	3.7×10^{-4}			0.09×10^{-6}
93	5	Convalex 10	None	50	71	-76	2×10^{-8}	6×10^{-9}	1.9×10^{-4}			0.04×10^{-6}
95	5	Convalex 10	None	25	75	-75	4×10^{-8}	3×10^{-8}	3.4×10^{-4}			0.07×10^{-6}
96	5	Convalex 10	None	50	69	-75	7×10^{-8}	6×10^{-9}	2.1×10^{-4}			0.05×10^{-6}
141	5	OS 124	212	50	75	-75	1×10^{-8}	5×10^{-9}	0.51×10^{-4}			0.015×10^{-6}

[a] 3-50% starts of the diffusion pump system with dry N$_2$ to atmospheric pressure.

[b] Oil was aerated.

[c] 2-100% starts with exposure to .01 torr.

Table 42

BAKEOUT AND RUN TIME VERSUS BACKSTREAMING

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp., °F	System Pressure, torr at Room Temp.	System Pressure, torr at LN$_2$ Temp.	Deposit Weight, mg/cm^2 Total	Deposit Weight, mg/cm^2 Analytical Blank	Net Backstreaming Deposit, mg/cm^2	Net Backstreaming Rate, mg/cm^2-min
139	1	Convalex 10	212	0	-75			0.9×10^{-4}	0.53×10^{-4}	0.9×10^{-4}	0.0518×10^{-6}
151	1	Convalex 10	473	0	-75			0.56×10^{-4}		0.03×10^{-4}	0.024×10^{-6}
114	1	Convalex 10	None	73.2	-75	6×10^{-8}	9×10^{-9}	2.3×10^{-4}	0.35×10^{-4}	2.3×10^{-4}	0.024×10^{-6}
152	1	Convalex 10	None	72	-77	3×10^{-7}	6×10^{-9}	1.4×10^{-4}		1.05×10^{-4}	0.024×10^{-6}
117	1	Convalex 10	212	73.7	-78	8×10^{-8}	6×10^{-9}	1.06×10^{-4}		1.06×10^{-4}	0.024×10^{-6}
208	1	Convalex 10	212	512	-60	3×10^{-7}	5×10^{-10}	1.27×10^{-4}		1.27×10^{-4}	0.0042×10^{-6}
218	1	Convalex 10	212	498	-75	1×10^{-7}	1×10^{-9}	1.02×10^{-4}		1.02×10^{-4}	0.0035×10^{-6}
202	1	Convalex 10	212	306	-75	5×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.13×10^{-4}	0.89×10^{-4}	0.0048×10^{-6}
184	1	Convalex 10	212	292	-73	4×10^{-7}	9×10^{-10}	1.04×10^{-4}	0.25×10^{-4}	0.79×10^{-4}	0.0045×10^{-6}
137	2	DC 705	212	0				3.2×10^{-4}		3.2×10^{-4}	
142	2	DC 705	464	0				2.5×10^{-4}		2.5×10^{-4}	
143	2	DC 705	500	0				1.9×10^{-4}		1.9×10^{-4}	
104	2	DC 705	RT	69.7	-74	3×10^{-8}	2×10^{-8}	6.3×10^{-4}		6.3×10^{-4}	0.150×10^{-6}
118	2	DC 705	212	73.7	-78	6×10^{-9}	1×10^{-9}	1.9×10^{-4}	0.80×10^{-4}	1.9×10^{-4}	0.043×10^{-6}
161	2	DC 705	212	73.7	-76	9×10^{-9}	1×10^{-9}	2.53×10^{-4}		1.73×10^{-4}	0.039×10^{-6}
107	2	DC 705	RT	151	-77	2×10^{-8}	2×10^{-8}	5.0×10^{-4}	1.6×10^{-4}	5.0×10^{-4}	0.056×10^{-6}
153	2	DC 705	RT	146	-77	4×10^{-9}	7×10^{-10}	3.2×10^{-4}		1.6×10^{-4}	0.018×10^{-6}
149	2	DC 705	212	145	-77	4×10^{-9}	9×10^{-10}	3.2×10^{-4}	2.2×10^{-4}	1.0×10^{-4}	0.011×10^{-6}
108	2	DC 705	RT	264	-75	6×10^{-9}	2×10^{-9}	6.5×10^{-4}	0.94×10^{-4}	6.5×10^{-4}	0.041×10^{-6}
157	2	DC 705	212	292.5	-74	5×10^{-9}	5×10^{-10}	1.9×10^{-4}		0.96×10^{-4}	0.0054×10^{-6}
172	2	DC 705	212	292	-77	5×10^{-9}	8×10^{-10}	3.2×10^{-4}	1.6×10^{-4}	1.6×10^{-4}	0.009×10^{-6}
207	2	DC 705	212	512	-60	3×10^{-9}	7×10^{-10}	3.2×10^{-4}	2.4×10^{-4}	3.2×10^{-4}	0.010×10^{-6}
219	2	DC 705	212	486	-75	3×10^{-9}	7×10^{-10}	4.75×10^{-4}		2.35×10^{-4}	0.008×10^{-6}

Table 42 (Cont.)

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp., °F	System Pressure, torr at Room Temp.	System Pressure at LN$_2$ Temp.	Deposit Weight, mg/cm^2 Total	Analytical Blank	Net Backstreaming Deposit, mg/cm^2	Rate, mg/cm^2-min
128[a]	3	Convalex 10	212	73.7	-75	5×10^{-9}	2×10^{-9}	6.2×10^{-4}		6.2×10^{-4}	0.141×10^{-6}
235	3	Convalex 10	212	76	-75(cs)	8×10^{-9}	2×10^{-9}	7.5×10^{-4}	0.13×10^{-4}	7.37×10^{-4}	0.158×10^{-6}
238	3	Convalex 10	212	67	-75(cs)	8×10^{-9}	2×10^{-9}	7.5×10^{-4}	0.28×10^{-4}	7.22×10^{-4}	0.180×10^{-6}
197[b]	3	Convalex 10	RT	159.5	-74(cs)	1×10^{-8}	7×10^{-9}	73.3×10^{-4}	7.4×10^{-4}	65.9×10^{-4}	0.765×10^{-6}
182	3	Convalex 10	212	148.5	-76(cs)	9×10^{-9}	1×10^{-9}	30.2×10^{-4}	0.20×10^{-4}	30.0×10^{-4}	0.338×10^{-6}
273	3	Convalex 10	212	146	-75(cs)	5×10^{-9}	2×10^{-9}	7.41×10^{-4}	0.25×10^{-4}	7.16×10^{-4}	0.082×10^{-6}
233	3	Convalex 10	212	150	-75(cs)	4×10^{-9}	2×10^{-9}	9.5×10^{-4}	0.23×10^{-4}	9.27×10^{-4}	0.102×10^{-6}
243	3	Convalex 10	212	167	-75	9×10^{-9}	4×10^{-9}	9.9×10^{-4}	0.26×10^{-4}	9.64×10^{-4}	0.096×10^{-6}
169	3	Convalex 10	212	290	-78(cs)	9×10^{-9}	2×10^{-9}	21.9×10^{-4}	0.24×10^{-4}	21.66×10^{-4}	0.125×10^{-6}
177	3	Convalex 10	212	313	-76(cs)	7×10^{-9}	2×10^{-9}	41.2×10^{-4}	0.31×10^{-4}	40.89×10^{-4}	0.207×10^{-6}
186	3	Convalex 10	212	292	-73(cs)	5×10^{-9}	9×10^{-10}	49.2×10^{-4}	0.24×10^{-4}	48.96×10^{-4}	0.280×10^{-6}
216	3	Convalex 10	212	293	-75(cs)	3×10^{-9}	2×10^{-9}	12.0×10^{-4}	0.15×10^{-4}	11.85×10^{-4}	0.067×10^{-6}
228	3	Convalex 10	212	249.5	-25(cs)	5×10^{-9}	2×10^{-9}	13.7×10^{-4}	0.15×10^{-4}	13.55×10^{-4}	0.090×10^{-6}
210[c]	3	Convalex 10	212	148	-68/10 (cs)	2×10^{-8}	2×10^{-9}	0.92×10^{-4}		0.92×10^{-4}	0.010×10^{-6}
205	3	Convalex 10	212	411	-44	2×10^{-8}	3×10^{-9}	5.4×10^{-4}	0.17×10^{-4}	5.23×10^{-4}	0.021×10^{-6}
127	4[d]	DC 705	212	0	-75	4×10^{-8}		15.6×10^{-4}		15.6×10^{-4}	0.333×10^{-6}
132	4[d]	DC 705	212	0	-76			14.1×10^{-4}		14.1×10^{-4}	0.493×10^{-6}
*	4[d]	DC 705	212	0	-			(20×10^{-4})		(20×10^{-4})	0.542×10^{-6}
111	4[d]	DC 705	RT	96	-75	1×10^{-8}	1×10^{-9}	19.1×10^{-4}		19.1×10^{-4}	0.412×10^{-6}
119	4[d]	DC 705	212	73.7	-75	7×10^{-9}	2×10^{-9}	21.8×10^{-4}		21.8×10^{-4}	0.364×10^{-6}
123	4[d]	DC 705	212	73.7	-75	4×10^{-9}	3×10^{-10}	23.9×10^{-4}		23.9×10^{-4}	0.542×10^{-6}
129	4[d]	DC 705	212	75	-76	2×10^{-9}	9×10^{-10}	18.5×10^{-4}		18.5×10^{-4}	0.408×10^{-6}
134	4[e]	DC 705	212	74	-193	5×10^{-9}	1×10^{-9}	18.3×10^{-4}	2.17×10^{-4}	16.13×10^{-4}	0.293×10^{-6}
155	4[e]	DC 705	RT	55.5	-193	9×10^{-9}	6×10^{-9}	17.8×10^{-4}	4.3×10^{-4}	13.5×10^{-4}	0.408×10^{-6}
163	4[e]	DC 705	RT	95	-76	6×10^{-8}	4×10^{-8}	20.7×10^{-4}	4.8×10^{-4}	15.9×10^{-4}	0.314×10^{-6}
176	4[f]	DC 705	212	290	-74	7×10^{-8}	2×10^{-8}	61.0×10^{-4}	6.5×10^{-4}	54.5×10^{-4}	0.293×10^{-6}
187	4[f]	DC 705	212	200	-76	6×10^{-8}	2×10^{-8}	32.6×10^{-4}	7.4×10^{-4}	25.2×10^{-4}	0.211×10^{-6}
231	4[f]	DC 705	212	79.5	-75	4×10^{-8}	3×10^{-8}	11.2×10^{-4}	2.8×10^{-4}	8.4×10^{-4}	0.174×10^{-6}
236	4[f]	DC 705	212	126	-75	4×10^{-8}	4×10^{-8}	16.1×10^{-4}	3.6×10^{-4}	12.5×10^{-4}	0.166×10^{-6}
230	4[g]	DC 705	212	150	-75	5×10^{-8}	7×10^{-8}	24.6×10^{-4}	6.5×10^{-4}	18.1×10^{-4}	0.199×10^{-6}
237	4[g]	DC 705	212	67	-75	2×10^{-7}	2×10^{-9}	3.48×10^{-4}	0.79×10^{-4}	2.69×10^{-4}	0.066×10^{-6}
240	4[g]	DC 705	212	71	-75	3×10^{-7}	1×10^{-9}	3.31×10^{-4}	1.26×10^{-4}	2.05×10^{-4}	0.048×10^{-6}
244	4[g]	DC 705	212	167	-75	7×10^{-7}	2×10^{-9}	3.49×10^{-4}		3.49×10^{-4}	0.035×10^{-6}

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Table 42 (Cont.)

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp., °F	System Pressure, torr		Deposit Weight, mg/cm^2		Net Backstreaming	
						at Room Temp.	at LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm^2	Rate, mg/cm^2-min
158	5	OS 124	RT	73.2	-76	9×10^{-9}	3×10^{-9}	1.4×10^{-4}	0.7×10^{-4}	0.7×10^{-4}	0.016×10^{-6}
165	5	OS 124	212	75	-75	1×10^{-8}	4×10^{-9}	0.7×10^{-4}	0.36×10^{-4}	0.35×10^{-4}	0.006×10^{-6}
141	5	OS 124	212	75.2	-75	1×10^{-9}	5×10^{-9}	0.51×10^{-4}	-	0.51×10^{-4}	0.015×10^{-6}
183	5	OS 124	212	148.5	-76	5×10^{-9}	4×10^{-9}	0.92×10^{-4}	0.25×10^{-4}	0.67×10^{-4}	0.008×10^{-6}
211	5	OS 124	212	148	-68/-10	2×10^{-7}	2×10^{-7}	1.15×10^{-4}	-	1.15×10^{-4}	0.013×10^{-6}
170	5	OS 124	212	290	-78	4×10^{-9}	2×10^{-9}	1.10×10^{-4}	0.10×10^{-4}	0.10×10^{-4}	0.006×10^{-6}
188	5	OS 124	212	294	-75	3×10^{-9}	1×10^{-9}	1.30×10^{-4}	0.25×10^{-4}	1.05×10^{-4}	0.006×10^{-6}
193	5	OS 124	212	293	-74	6×10^{-8}	5×10^{-8}	1.36×10^{-4}	0.46×10^{-4}	0.90×10^{-4}	0.005×10^{-6}
229	5	OS 124	212	274	-25	2×10^{-7}	2×10^{-7}	2.10×10^{-4}	0.13×10^{-4}	1.97×10^{-4}	0.012×10^{-6}

[a] NRC HN-6 Baffle had a leak in it.
[b] Started at 100% heat load - no steam.
[c] Chevron RC-61 Baffle warm and cold sides.
[d] Station had BC-61 chevron baffle with cold sides.
[e] Station had G/P cyrosorb trap 2.
[f] Station had G/P cyrosorb trap 1.
[g] Station had a right-angle elbow baffle.
[h] Seeding Experiment: 25.0×10^{-4} mg/cm^2 rinsed off = 23.9×10^{-4}
25.0×10^{-4} mg/cm^2 steamed overnight has 17.7×10^{-4} mg/cm^2 on plate.

Table 43

EFFECT OF COOLING BAFFLE WALLS

Run	Station	Baffle	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp. and Sides Temp., °F	System Pressure, torr at Room Temp.	System Pressure, torr at LN₂ Temp.	Deposit Weight, mg/cm² Total	Deposit Weight, mg/cm² Analytical Blank	Net Backstreaming Deposit, mg/cm²	Net Backstreaming Rate, mg/cm²-min
100	1	BC-61	Convalex 10	None	70	-76/80	1×10^{-7}	4×10^{-9}	4.0×10^{-4}			0.10×10^{-6}
103	1	BC-61	Convalex 10	None	74.2	-76/80	5×10^{-8}	6×10^{-9}	2.4×10^{-4}			0.04×10^{-6}
109	1	BC-61	Convalex 10	212	168	-75/80	1×10^{-7}	7×10^{-8}	1.9×10^{-4}			0.02×10^{-6}
113	1	BC-61	Convalex 10	None	170	-78/80	6×10^{-8}	6×10^{-9}	4.2×10^{-4}			0.04×10^{-6}
117	1	BC-61	Convalex 10	212	73.7	-78/14	8×10^{-8}	6×10^{-9}	1.06×10^{-4}			0.024×10^{-6}
121	1	BC-61	Convalex 10	212	73.7	-78/14	7×10^{-7}	9×10^{-9}	1.06×10^{-4}			0.024×10^{-6}
152	1	BC-61	Convalex 10	None	72	-77/14	3×10^{-7}	7×10^{-9}	1.40×10^{-4}	0.35×10^{-4}	1.05×10^{-4}	0.024×10^{-6}
160	1	BC-61	Convalex 10	212	75.7	-75/14	2×10^{-7}	9×10^{-9}	0.80×10^{-4}	0.26×10^{-4}	0.54×10^{-4}	0.012×10^{-6}
202	1	BC-61	Convalex 10	212	306	-76/14	5×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.13×10^{-4}	0.89×10^{-4}	0.004×10^{-6}
208	1	BC-61	Convalex 10	212	512	-60/14	3×10^{-7}	5×10^{-10}	1.27×10^{-4}	0.00	1.27×10^{-4}	0.004×10^{-6}
98	2	BC-61	DC-705	None	74	-76/80	5×10^{-8}	3×10^{-8}	19.2×10^{-4}			0.477×10^{-6}
101	2	BC-61	DC-705	None	71	-76/80	5×10^{-8}	3×10^{-8}	18.3×10^{-4}			0.427×10^{-6}
108	2	BC-61	DC-705	None	264	-75/14	6×10^{-9}	2×10^{-9}	6.5×10^{-4}			0.041×10^{-6}
149	2	BC-61	DC-705	212	145	-77/14	4×10^{-9}	9×10^{-9}	3.2×10^{-4}	2.2×10^{-4}	1.0×10^{-4}	0.011×10^{-6}
161	2	BC-61	DC-705	212	73.7	-76/14	9×10^{-9}	1×10^{-9}	2.53×10^{-4}	0.80×10^{-4}	1.73×10^{-4}	0.039×10^{-6}
181	2	BC-61	DC-705	212	75	-76/14	7×10^{-9}	1×10^{-10}	1.57×10^{-4}	1.57×10^{-4}	0.00×10^{-4}	–
203	2	BC-61	DC-705	212	306	-76/14	3×10^{-9}	6×10^{-10}	7.6×10^{-4}	1.57×10^{-4}	6.03×10^{-4}	0.033×10^{-6}
207	2	BC-61	DC-705	212	512	-60/14	3×10^{-9}	7×10^{-10}	3.17×10^{-4}	0.0	3.17×10^{-4}	0.010×10^{-6}
205	3	BC-61	Convalex 10	212	411	-44/80	2×10^{-8}	3×10^{-9}	5.40×10^{-4}	0.17×10^{-4}	5.23×10^{-4}	0.021×10^{-6}
210	3	BC-61	Convalex 10	212	148	-25/20	2×10^{-8}	2×10^{-9}	0.92×10^{-4}	0.0	0.92×10^{-4}	0.010×10^{-6}
243	3	HN-6	Convalex 10	212	167	-75/80	9×10^{-9}	4×10^{-9}	9.90×10^{-4}	0.26×10^{-4}	9.64×10^{-4}	0.096×10^{-6}
233	3	HN-6	Convalex 10	212	77	-75/14	8×10^{-9}	2×10^{-9}	7.50×10^{-4}	0.13×10^{-4}	7.37×10^{-4}	0.158×10^{-6}
238	3	HN-6	Convalex 10	212	67	-75/14	8×10^{-9}	2×10^{-9}	7.50×10^{-4}	0.28×10^{-4}	7.22×10^{-4}	0.180×10^{-6}

Table 44

EFFECT OF LIGHT END REMOVAL

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp. and Sides Temp., °F	System Pressure, torr at Room Temp.	System Pressure, torr at LN_2 Temp.	Deposit Weight, mg/cm² Total	Deposit Weight, mg/cm² Analytical Blank	Net Backstreaming Deposit, mg/cm²	Net Backstreaming Rate, mg/cm²-min
67	1	Convalex 10	None	17	RT	2×10^{-5}	2×10^{-8}	39.9×10^{-4}			3.9×10^{-6}
69	1	Convalex 10	None	21	RT/185	7×10^{-6}	5×10^{-8}	23.7×10^{-4}			1.87×10^{-6}
71	1	Convalex 10	None	67	RT/185	5×10^{-6}	7×10^{-8}	113.5×10^{-4}			2.82×10^{-6}
74	1	Convalex 10	None	23	RT/185	4×10^{-6}	2×10^{-8}	12.1×10^{-4}			0.88×10^{-6}
76	1	Convalex 10	None	22	RT/190	3×10^{-6}	5×10^{-7}	14.6×10^{-4}			1.1×10^{-6}
81	1	Convalex 10	None	115	RT/180	7×10^{-7}	5×10^{-7}	72.8×10^{-4}			1.05×10^{-6}
84	1	Convalex 10	None	24	RT	9×10^{-7}	5×10^{-7}	16.6×10^{-4}			1.17×10^{-6}
146	1	Convalex 10	None	73.7	RT	4×10^{-7}	1×10^{-7}	44.1×10^{-4}			1.0×10^{-6}
145	2	DC-705	None	73.7	RT	3×10^{-8}	2×10^{-9}	81.8×10^{-4}	2.2×10^{-4}	49.5×10^{-4}	1.85×10^{-6}
234	2	DC-704/705	212	77	RT	1×10^{-8}	2×10^{-9}	51.7×10^{-4}			1.07×10^{-6}
61	3	Convalex 10	None	120	RT	2×10^{-6}	5×10^{-8}	213.0×10^{-4}			2.96×10^{-6}
66	3	Convalex 10	None	26.5	RT	1×10^{-7}		55.5×10^{-4}			3.5×10^{-6}
68	3	Convalex 10	None	17	RT	1×10^{-7}		70.0×10^{-4}			0.685×10^{-6}
70	3	Convalex 10	None	22	RT	2×10^{-7}	6×10^{-8}	46.2×10^{-4}			3.47×10^{-6}
73	3	Convalex 10	None	65.5	RT	1×10^{-7}	3×10^{-8}	33.4×10^{-4}			0.85×10^{-6}
75	3	Convalex 10	None	23	RT	1×10^{-7}		46.6×10^{-4}			3.38×10^{-6}
78	3	Convalex 10	None	20	RT	7×10^{-8}	2×10^{-8}	11.8×10^{-4}			0.98×10^{-6}
164	3	Convalex 10	None	75	RT	1×10^{-7}	1×10^{-8}	10.7×10^{-4}	0.55×10^{-4}	11.15×10^{-4}	0.224×10^{-6}
110	4	DC-705	None	391	RT	6×10^{-9}	1×10^{-9}	106×10^{-4}			0.454×10^{-6}
124	4	DC-705	None	73.5	RT	4×10^{-8}	2×10^{-9}	174×10^{-4}			3.96×10^{-6}
154	4	DC-705	None	117	RT	5×10^{-8}		68.5×10^{-4}	14.8×10^{-4}	53.7×10^{-4}	0.770×10^{-6}
168	4	DC-705	None	240	RT	2×10^{-8}	4×10^{-9}	211.0×10^{-4}	6.5×10^{-4}	204.5×10^{-4}	1.424×10^{-6}
105	5	Convalex 10	None	67.2	RT	4×10^{-8}	2×10^{-8}	5.70×10^{-4}			0.142×10^{-6}
138	5	OS 124	None	1507	RT	3×10^{-8}	6×10^{-9}	3.83×10^{-4}	0.25×10^{-4}	5.15×10^{-4}	0.004×10^{-6}
147	5	OS 124	None	73.7	RT			5.40×10^{-4}			0.118×10^{-6}

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Table 45

EFFECT OF BAFFLE TEMPERATURE ON BACKSTREAMING
(EXCLUDING LIGHT END REMOVAL)

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp. and Sides Temp., °F	System Pressure, torr		Deposit Weight, mg/cm²		Net Backstreaming	
						at Room Temp.	at LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
202	1	Convalex 10	212	306	-76	4×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.13×10^{-4}	0.89×10^{-4}	0.004×10^{-6}
208	1	Convalex 10	212	512	-60	3×10^{-7}	5×10^{-10}	1.27×10^{-4}		1.27×10^{-4}	0.004×10^{-6}
212	1	Convalex 10	212	135	-23	6×10^{-7}	1×10^{-9}	1.30×10^{-4}	0.65×10^{-4}	0.65×10^{-4}	0.008×10^{-6}
185	2	DC-705	212	291	-73	4×10^{-9}	7×10^{-10}	4.73×10^{-4}	3.17×10^{-4}	1.56×10^{-4}	0.009×10^{-6}
207	2	DC-705	212	512	-60	3×10^{-9}	7×10^{-10}	3.17×10^{-4}	0.30×10^{-4}	2.87×10^{-4}	0.010×10^{-6}
219	2	DC-705	212	486	-75	3×10^{-8}	7×10^{-9}	4.75×10^{-4}	2.40×10^{-4}	2.35×10^{-4}	0.008×10^{-6}
213	2	DC-705	212	247	-23	3×10^{-9}	1×10^{-9}	4.00×10^{-4}	1.90×10^{-4}	2.10×10^{-4}	0.014×10^{-6}
225	2	DC-705	212	126	-75	5×10^{-9}	1×10^{-9}	2.85×10^{-4}		2.85×10^{-4}	0.038×10^{-6}
227	2	DC-705	212	148	-25	7×10^{-9}	1×10^{-9}	3.17×10^{-4}	0.79×10^{-4}	2.38×10^{-4}	0.027×10^{-6}
216	3	Convalex 10	212	293	-75	3×10^{-9}	2×10^{-9}	12.00×10^{-4}	0.15×10^{-4}	11.85×10^{-4}	0.065×10^{-6}
228	3	Convalex 10	1212	249.5	-25	5×10^{-9}	2×10^{-9}	13.70×10^{-4}	0.15×10^{-4}	13.55×10^{-4}	0.090×10^{-6}
210	3	Convalex 10	212	148	-23	2×10^{-8}	2×10^{-9}	0.92×10^{-4}		0.92×10^{-4}	0.010×10^{-6}
183	5	OS 124	212	148.5	-76	5×10^{-9}	4×10^{-9}	0.92×10^{-4}	0.25×10^{-4}	0.67×10^{-4}	0.008×10^{-6}
211	5	OS 124	212	148	-25	2×10^{-7}	2×10^{-7}	1.15×10^{-4}		1.15×10^{-4}	0.013×10^{-6}
170	5	OS 124	212	290	-78	4×10^{-9}	2×10^{-9}	1.10×10^{-4}	0.10×10^{-4}	1.00×10^{-4}	0.006×10^{-6}
229	5	OS 124	212	274	-25	2×10^{-7}	2×10^{-7}	2.10×10^{-4}	0.13×10^{-4}	1.97×10^{-4}	0.012×10^{-6}

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Table 46

EFFECT OF LIQUID NITROGEN COOLING OF CRYOSORB BAFFLE ON BACKSTREAMING

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp., °F	System Pressure, torr		Deposit Weight, mg/cm²		Net Backstreaming	
						at Room Temp.	at LN₂ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
155	4	DC-705	RT	55.5	LN$_2$	9×10^{-9}	6×10^{-9}	17.8×10^{-4}	4.3×10^{-4}	13.5×10^{-4}	0.408×10^{-6}
159	4	DC-705	RT	51	LN$_2$		6×10^{-9}	21.5×10^{-4}	10.8×10^{-4}	10.7×10^{-4}	0.349×10^{-6}
163	4	DC-705	RT	95	LN$_2$	6×10^{-9}	4×10^{-9}	20.7×10^{-4}	4.8×10^{-4}	15.9×10^{-4}	0.293×10^{-6}
168	4	DC-705	RT	240	RT	2×10^{-8}	4×10^{-9}	211.0×10^{-4}	6.5×10^{-4}	204.5×10^{-4}	1.424×10^{-6}
176	4	DC-705	RT	290	-76	7×10^{-8}	2×10^{-8}	61.0×10^{-4}	6.5×10^{-4}	54.5×10^{-4}	0.314×10^{-6}
187	4	DC-705	212	200	-74	6×10^{-8}	2×10^{-8}	32.6×10^{-4}	7.4×10^{-4}	25.2×10^{-4}	0.211×10^{-6}
230	4	DC-705	212	150	-75	5×10^{-8}	9×10^{-8}	24.6×10^{-4}	6.5×10^{-4}	19.1×10^{-4}	0.199×10^{-6}
231	4	DC-705	212	80	-75	4×10^{-8}	3×10^{-9}	11.2×10^{-4}	2.8×10^{-4}	8.4×10^{-4}	0.174×10^{-6}
236	4	DC-705	212	126	-75	4×10^{-8}	4×10^{-9}	16.1×10^{-4}	3.6×10^{-4}	12.5×10^{-4}	0.166×10^{-6}

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Table 47

EFFECTS ON LEAKS IN THE FINE SIDE ON BACKSTREAMING

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp., °F	Fore Pressure, μ	System Pressure, torr		Deposit Weight, mg/cm²		Net Backstreaming	
							at Room Temp.	at LN₂ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²·min
224[a]	1	Convalex 10	212	126	-75	8		6.0×10^{-6}	1.35×10^{-4}		1.35×10^{-4}	0.0179×10^{-6}
166[b]	1	Convalex 10	212	290	-75	8	3×10^{-7}	6×10^{-8}	0.75×10^{-4}	0.05×10^{-4}	0.70×10^{-4}	0.0041×10^{-6}
203[c]	1	Convalex 10	212	306	-75	8	5×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.13×10^{-4}	0.89×10^{-4}	0.0048×10^{-6}
180[c]	1	Convalex 10	212	75	-75	8	1×10^{-7}	1×10^{-9}	1.02×10^{-4}		1.02×10^{-4}	0.0227×10^{-6}

[a] With oxygen in the fine vacuum side P_{LN_2} = 6 × 10⁻⁶ torr. After 30 min, no leak, P_{LN_2} = 1.6 × 10⁻⁸ torr.

[b] With air in the fine vacuum side P_{LN_2} = 6 × 10⁻⁸ torr. After 30 min, no leak, P_{LN_2} = 2.1 × 10⁻⁹ torr.

[c] These runs represent the average low backstreaming values.

Table 48

EFFECT OF LEAKS IN THE FORE LINE ON BACKSTREAMING

Run	Station	Oil	Bakeout Temp., °F	Length of Run, hr	Trap Temp., °F	Fore Pressure, μ	System Pressure, torr		Deposit Weight, mg/cm²		Net Backstreaming	
							at Room Temp.	at LN$_2$ Temp.	Total	Analytical Blank	Deposit, mg/cm²	Rate, mg/cm²-min
222[a]	5	OS 124	212	146	-75	400	5 →20 x 10^{-7}		19.2 x 10^{-4}	0.15 x 10^{-4}	19.05 x 10^{-4}	0.214 x 10^{-6}
175[b]	5	OS 124	212	313	-75	50	5 x 10^{-9}	4 x 10^{-9}	1.02 x 10^{-4}	0.0	1.02 x 10^{-4}	0.0059 x 10^{-6}
193[c]	5	OS 124	212	292	-74	5	6 x 10^{-8}	5 x 10^{-8}	1.36 x 10^{-4}	0.46 x 10^{-4}	0.90 x 10^{-4}	0.0052 x 10^{-6}
183[c]	5	OS 124	212	148.5	-76	4	5 x 10^{-9}	4 x 10^{-9}	0.92 x 10^{-4}	0.25 x 10^{-4}	0.63 x 10^{-4}	0.0075 x 10^{-6}

[a] Fore line pressure 400 μ by TIC gauge, pulsing of pressure in fine vacuum.

[b] Fore line pressure 50 μ by TIC gauge.

[c] Two runs to compare deposit and time with no leak in fore line.

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Table 49

GAS INJECTION STUDIES

Date	Gas	Leak S/ΔP[a]	Leak Location[b]	Fore Pressure, torr[c] TC	Fore Pressure, torr[c] McLeod	Pressure[d] System	Pressure[d] Gas	PPA, amp	Apparent CFe
7/29	O		4	3	5	1.9×10^{-7}	1.4×10^{-10} S	2×10^{-12}	
	He		4	11	14	1.8×10^{-7}	1.4×10^{-10} S	2×10^{-12}	
	He		4	41	70	1.8×10^{-7}	1.4×10^{-10} S	2×10^{-12}	
	He		4	40	63	1.8×10^{-7}	1.4×10^{-10} S	2×10^{-12}	
	He		4	53	83	1.7×10^{-7}	1.4×10^{-10} S	2×10^{-12}	6×10^{7}
7/30	O		4	7	8	1.7×10^{-7}	1.4×10^{-10} S	2×10^{-12}	
	He		4	93	150	1.65×10^{-7}	3.5×10^{-10} S	5×10^{-12}	4×10^{8}
	He		4	140	220		7×10^{-9}	1×10^{-11}	
	He		4	200	325		5.6×10^{-8} S	8×10^{-11}	
	He		4	290	440	1.7×10^{-7}	2.7×10^{-7} S	3×10^{-9}	
	He		4	350	530	2.05×10^{-7}	2.0×10^{-7} P	3.5×10^{-8}	2×10^{6}
	He		4	420	630	$0.8\text{-}1.4 \times 10^{-6}$		$1\text{-}1.5 \times 10^{-8}$	3×10^{6}
7/31	O		-	8	10	1.75×10^{-7}	1.4×10^{-10} S	2×10^{-12}	
	He		4	310	480	1.9×10^{-7}	9×10^{-9} S	1.3×10^{-8}	
	He		4	380	530	2.3×10^{-7}	3.1×10^{-7} P	3.5×10^{-8}	2×10^{6}
9/23	O			3×10^{-3}		6.8×10^{-8}			
	He	65/48 (1.1)	2		(10×10^{-3})	7.0×10^{-7}	3.9×10^{-6} P	1×10^{-7}	2×10^{3}
	He	70/48 (9.7)	2		(10×10^{-3})	4.2×10^{-6}	2.3×10^{-5} P	8.3×10^{-8}	5×10^{2}
	He	74/48 (15)	2		(10×10^{-3})	9.5×10^{-6}	5.3×10^{-5} P	3×10^{-8}	2×10
9/24	O			3×10^{-3}		1.8×10^{-8}		6.8×10^{-10}	
	He	60/50 (0.03)	3	3×10^{-3}	(10×10^{-3})	2×10^{-8}	1.7×10^{-9} S	7.0×10^{-10}	6×10^{6}
	He	65/50 (1.1)	3	4×10^{-3}	(10×10^{-3})	1.3×10^{-6}	2×10^{-6} P	6.5×10^{-7}	5×10^{3}
	He	70/50 (10)	3	7×10^{-3}	(10×10^{-3})	9.2×10^{-6}	5×10^{-5} P	7.5×10^{-6}	10^{3}
9/29	O			3×10^{-3}		1.8×10^{-8}		6.8×10^{-10}	
	H₂	60/50	2	3×10^{-3}	(10×10^{-3})	2×10^{-8}	1.7×10^{-9} S	7.0×10^{-10}	6×10^{6}
	H₂	65/50 (0.3)	2	3×10^{-3}	(10×10^{-3})	1.3×10^{-6}	2×10^{-6} P	6.5×10^{-7}	5×10^{3}
	H₂	70/50 (1.7)	2	3×10^{-3}	(10×10^{-3})	9.2×10^{-6}	5×10^{-5} P	7.5×10^{-6}	2×10^{2}
9/29	O			3×10^{-3}		1.9×10^{-8}			
	H₂	60/50	3	3×10^{-3}	(10×10^{-3})	2.3×10^{-8}	1.6×10^{-8} S	6.5×10^{-10}	6×10^{5}
	H₂	65/50 (0.3)	3	3×10^{-3}	(10×10^{-3})	1.8×10^{-7}	3×10^{-6} P	1×10^{-7}	3×10^{4}
	H₂	70/50 (1.7)	3	3×10^{-3}	(10×10^{-3})	3.3×10^{-6}	6×10^{-6} P	2.2×10^{-6}	2×10^{8}

[a] Signifies an undefined leak. S/ΔP signifies valve setting/inlet gas pressure. Setting is not reproducible and used only for comparison. Estimated rate given as ml per min.

[b] Locations are: 2, between top and next lower jet; 3, liquid at bottom outside jet stack; 4, fore line ahead of TC gauge.

[c] Normally measured with thermocouple (TC) gauge. Absolute value of noncondensible measured with Lippincott-Tilting McLeod; lowest reading 1 mm = 1×10^{-3} torr.

[d] System pressure as read by ion gauge at RT. Injection gas pressure determined by differences in ion gauge reading using Varian gauge constants (P) or by mass spectrometer sensitivities (S) from Table 23.

[e] Apparent compressibility factor; fore pressure/system pressure.

Table 50

EFFECT OF TRAP CONDITIONS ON MASS
SPECTROMETER READINGS AT $m/e = 78$

Trap	amp/torr
Room-temperature zeolite[a]	1×10^{-12}
Liquid nitrogen[a]	None
25°C[a]	5.3×10^{-10}
-78°C[a]	6.3×10^{-10}
-196°C[a]	6.4×10^{-13}
25°C Zeolite[a]	1.1×10^{-10}
-78°C Zeolite[a]	1.8×10^{-12}
-196°C Zeolite[a]	8.4×10^{-14}
-75°F, Chevron[b]	1×10^{-12}
70°F, Chevron[b]	5×10^{-12}

[a] Bryant and Gosselin, DC 705, Ref. 4.

[b] This program, Convalex 10.

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Table 51

SENSITIVITIES FOR HELIUM[a]

Experiment	Leak Setting	B-A Gauge Helium Press., torr N_2 equiv)	Sensitivity, amps/torr	Experiment	Leak Setting	B-A Gauge Helium Press., torr N_2 equiv)	Sensitivity, amps/torr	Experiment	Leak Setting	B-A Gauge Helium Press., torr N_2 equiv)	Sensitivity, amps/torr
8/12	57.5/0	3×10^{-8}	.11	9/17	64/0	3.3×10^{-6}	.054	10/6	65/52	9.0×10^{-7}	.067
8/13	57/0	5×10^{-8}	.046		65	4.1×10^{-6}	.060		67.5/52	3.6×10^{-6}	.011
	58/0	2.1×10^{-7}	.041		66	5.0×10^{-6}	.067		70/52	9.0×10^{-6}	.010
	60/0	8.9×10^{-7}	.024		67	5.6×10^{-6}	.080		75/52	3.4×10^{-5}	.012
	61/0	1.3×10^{-6}	.028		68	6.1×10^{-6}	.084		77.5/52	9.0×10^{-5}	.011
	62/0	2.2×10^{-7}	.026		69	7.8×10^{-5}	.093		80/52	1.5×10^{-4}	.013
8/14	58/0	1.1×10^{-7}	.067		70	1.1×10^{-5}	.086		70/52	1.2×10^{-5}	.013
	59/0	2.7×10^{-7}	.067		71	1.2×10^{-5}	.083		65/52	2.6×10^{-6}	.012
	60/0	5.6×10^{-7}	.061	9/23	60/67	3.4×10^{-5}	.012		65.5/52	8.0×10^{-7}	.012
	61/0	8.4×10^{-7}	.060		65/66	1.4×10^{-5}	.014		60/52	3.4×10^{-8}	.010
	62/0	1.2×10^{-6}	.072		70/63	1.2×10^{-4}	.021		72.5/52	1.6×10^{-5}	.030
	63/0	1.7×10^{-6}	.063		74/56	1.7×10^{-4}	.053	10/5	60/52	3.0×10^{-7}	.013
	64/0	2.1×10^{-6}	.071		74/52	1.2×10^{-4}	.063		62.5/52	9.0×10^{-6}	.0007
	65/0	3.0×10^{-6}	.074		65/48	3.9×10^{-6}	.026		65/52	2.1×10^{-6}	.006
	66/0	3.7×10^{-6}	.075		70/48	2.3×10^{-5}	.036		67.5/52	8.4×10^{-6}	.012
	67/0	5.0×10^{-6}	.077		74/48	5.3×10^{-5}	.057		70/52	1.4×10^{-5}	.011
	68/0	6.2×10^{-6}	.088	9/24	70/50	9.0×10^{-6}	.024		72.5/52	2.5×10^{-5}	.014
9/17	59/0	1.4×10^{-6}	.064		69/50	1.4×10^{-8}	.036		75/52	5.6×10^{-5}	.016
	60/0	1.6×10^{-6}	.065		60/50	5.6×10^{-7}	.009		77.5/52	1.2×10^{-4}	.016
	61/0	1.8×10^{-6}	.074		65/52	7.8×10^{-7}	.041		80/52	2.0×10^{-4}	.017
	62/0	2.2×10^{-6}	.073		70/52	1.0×10^{-5}	.035		82.5/52	3.4×10^{-4}	.017
	63/0	2.7×10^{-6}	.069		65/52	1.3×10^{-6}	.040				.019

[a]Dynode voltage setting 2000, electron voltage 70, emission current 1.0 mamp. Based on helium pressure as determined by Bayard-Alpert Gauge.

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Table 52

SENSITIVITIES[a] FOR NITROGEN, OXYGEN, WATER, METHANOL, AND BENZENE

Date	Leak Setting	Magnet	B-A Gauge Pressure, torr	Leaked Gas	Sensitivity, amp/torr				
					Nitrogen (28)	Oxygen (32)	Water (18)	Methanol (31)	Benzene (28)
7/22	-	S	2.1×10^{-5}	Nitrogen	.048				
7/21	-	S	2.0×10^{-5}	Nitrogen	.066				
7/22	-	S	2.1×10^{-5}	Nitrogen	.062				
7/22	-	S	1.9×10^{-5}	Nitrogen	.17				
7/23	-	S	3.1×10^{-5}	Oxygen		.0082			
7/22	-	S	3.7×10^{-5}	Oxygen		.011			
11/19	70/0	S	[b]3.2×10^{-7}	Air	.10	.057			
10/13	75/0	S	8.5×10^{-7}	Air	.055	.031	.22		
11/20	77/0	S	[b]8.0×10^{-6}	Air	.070	.060	.17	.36	
11/20	77/0	S	[b]1.0×10^{-5}	Air	.051	.062	.20		.000013
10/19	75/0	L	2.8×10^{-6}	Air	.045	.034	.11		
10/26	75/0	L	3.0×10^{-6}	Air	.17	.13	.17		
10/26	80/0	L	1.0×10^{-6}	Air	.11	.08	.08		
10/26	70/0	L	6.7×10^{-7}	Air	.093	.063	.16		
10/14	75/0	L	7.7×10^{-7}	Air	.057	.050	.066		
10/15	80/0	L	2.6×10^{-5}	Air	.097	.073	.13		.016
10/16	77.5/0	L	1.5×10^{-5}	Air	.083	.070	.23		.022
10/16	75/0	L	6.0×10^{-6}	Air	.068	.039	.28		.014
10/14	80/0	L	4.8×10^{-6}	Air	.13	.083	.11		.011
11/20	77/0	L	[b]1.0×10^{-5}	Air	.11	.10	.35		.003
11/20	77/0	L	[b]1.1×10^{-5}	Air	.092	.078			.007

[a] Dynode voltage 2000, electron voltage 70, emission current 1.0 mamp.

[b] Bayard-Alpert gauge trapped with liquid nitrogen. This lowered the room temperature reading 10 to 20%.

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Table 53

BENZENE MASS FREQUENCY PATTERN

Mass	API	Relative Frequency[a] 11TR1
2		64[b]
12		9[b]
13		3.4[b]
15		57[b]
25	0.5	2
26	3.4	14
27	3.0	11
29		106[b]
30		7[b]
31		78[b]
37	4.1	7
37.5	1.3	–
38	5.8	11
39	14.2	36
49	2.5	5
50	15.7	32
51	18.6	36
52	19.4	36
53	8.4	–
63	2.8	5
73	1.5	–
74	4.6	8
75	1.6	–
76	6.0	–
77	14.0	–
78	100.0	100
79	6.0	–

[a]All determined with large magnet except mass 2.

[b]Values are not associated with benzene spectrum as such.

Table 54

SPECIFICATIONS OF TURBOMOLECULAR PUMP

Supplier: The Welch Scientific Company
                         Skokie, Illinois

Type: model 3102A, a skid-mounted self-contained
                      unit consisting of:

                         Turbomolecular pump with transmission
                         assembly
                         Self-contained refrigerator for bearing
                         cooling
                         Forepump is Duo-Seal vacuum pump No. 1397B
                         electrical control panel.

Power: 220 volt, 3-phase, 60 cycle using a maximum
                      starting current of 0.9 amp.

Capacity   Forepump is 15 cfm free air
(pumping speed)   425 liters/min
                      Turbomolecular pump 260 liters/min
                                          140 liters/min (with
                                          inlet protector screen)

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Table 55

STANDARD OPERATING CONDITIONS

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1. Nitrogen is bled into the fore line sufficient to maintain ~ 2 torr while forepump is started.

2. The turbomolecular pump is started with leak at 2 torr. After full turbine speed is reached, leak lowered to 1.1 torr.

3. When fine pressure is 10^{-4} torr range, turn off leak. Fore pressure goes to $< 10^{-3}$ torr.

4. Bakeout locations:
   a. Collection plate
   b. BC-61 baffle on Station 8
   c. Center of turbomolecular pump and throat.

5. Run time is 74 hr.

6. To terminate run, allow baffle and plate to warm up for 18 hr.

7. Helium is bled into fine side until pressure is 1 torr.

8. The turbomolecular pump is shut off; when pump stops, it is valved off from the mechanical pump. The fine side is let up to atmospheric pressure.

9. It is optional whether or not the forepump is turned off.

10. Sample as per IIIB 3, Part A.

Table 56

FINE PRESSURE VERSUS FORE PRESSURE

Fore-line Pressure (Mc Cloud gauge), torr	Fine Pressure (Varian gauge), torr
1.10[a]	2×10^{-4}
1.01[a]	4×10^{-5}
1.00	2×10^{-5}
0.097	8×10^{-6}
0.085	4×10^{-6}
0.060	3×10^{-6}
No leak	2×10^{-6}

[a]Repeated 3 times.

Table 57

CALIBRATION DATA FOR ULTRAVIOLET ABSORBANCE

Cell	Absorbance Band, mμ	Concentration, mg/ml	Absorbance units	Slope	Deviation, %
1 cm	252	0.996	0.910	1.09	.9
		0.747	0.670	1.12	2.8
		0.398	0.370	1.08	.0
		0.199	0.186	1.07	.9
		0.099	0.095	1.04	3.7
				Avg 1.08	Avg 1.7
2 cm	252	0.292	0.530	0.558	8.1
		0.146	0.262	0.552	7.0
		0.073	0.140	0.522	1.2
		0.044	0.080	0.548	6.2
		0.014	0.035	0.400	22.5
				Avg 0.516	Avg 9.0

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Table 58

ACCURACY OF ULTRAVIOLET ANALYTICAL METHODS FOR TURBOMOLECULAR OIL

Weight by Seeding, mg	mg/cm^2	Rinse Volume, ml	Absorbance Units	Total, mg/cm^2	Analytical Blank, cm/cm^2	Net Weight, mg/cm^2	% Error
1.069	72.5×10^{-4} [a]	10	0.093	68.4×10^{-4}		68.4×10^{-4} [b]	-5.66
0.712	48.4×10^{-4} [a]	10	0.062	45.5×10^{-4}		45.5×10^{-4} [b]	-6.00
0.356	24.2×10^{-4} [a]	10	0.032	23.5×10^{-4}		23.5×10^{-4} [b]	-2.90
0.170	11.6×10^{-4} [a]	10	0.018	13.2×10^{-4}		13.2×10^{-4}	13.8
0.170	11.6×10^{-4} [a]	10	0.024	17.6×10^{-4}	0×10^{-4}	13.9×10^{-4}	19.8
0.102	6.9×10^{-4} [a]	10	0.012	8.8×10^{-4}	3.70×10^{-4}	5.1×10^{-4}	-26.0
0.034	2.3×10^{-4} [a]	10	0	0	3.70×10^{-4}	0	

[a] Referred to a collection plate area of 147 cm^2.

[b] Measured in a 1-cm cell; others in a 2-cm cell.

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Table 59

GAS CHROMATOGRAPHIC DATA ON COMPARISON
OF UNUSED TURBOMOLECULAR PUMP OIL
VERSUS STRAIGHT-CHAIN HYDROCARBONS[a]

Hydrocarbon Standard	Retention Time		
	Standard	Unused Oil	Used Oil
C_{15}		10.5	10.5
		13.4	13.2
C_{16}	15.5	15.6	15.7
		19.8	19.8
C_{17}		24.0	24.5
		30.2	30.4
C_{18}	36.0	37.4	37.6
		46.7	47.7
C_{19}		56.9	58.0
		72.6	72.3
C_{20}		86.9	88.9
		104.6	108.2
C_{21}	132.0	133.8	

[a]Operating conditions: Loenco dual-
flame model 70; 6 ft x 3/16 in SE 30
3% columns; column temp., 165°C;
injection temp., 250°C; detector temp.,
260°C; helium pressure, 26 psi; and
sensitivity, 16X.

Table 60

GAS CHROMATOGRAPHIC DATA ON COMPARISON
OF USED TURBOMOLECULAR PUMP OIL
VERSUS STRAIGHT-CHAIN HYDROCARBONS[a]

Hydrocarbon	Retention Time	Sample μg	Area, cm^2	Relative Response Factor
C_{16}	15.5	4.92	12.45	2.53
C_{18}	36.0	6.96	15.60	2.25
C_{21}	132.0	11.58	20.65	1.78

Major Peak Recovery Based on 0.5 μliter Sample

Hydrocarbon	Retention Time	Area, cm^2	Relative Response Factor	Sample, μg/0.5 liter
C_{16}	15.6	2.57	2.53	1.02
	(19.8)	2.30	2.53	0.91
C_{17}	24.0	15.40	2.25	6.84
	(30.2)	5.82	2.25	2.59
C_{18}	37.4	17.92	2.25	7.96
	(46.7)	6.40	2.25	2.84
C_{19}	56.9	16.87	2.25	7.50
	(72.6)	6.88	1.78	3.87
	(86.9)	8.10	1.78	4.55
				38.08

$$\frac{38.1 \; \mu\text{g recovered}}{500 \; \mu\text{g sample}} \times 100 = 7.6\% \text{ recovered.}$$

[a] Operating conditions were the same as given in Table 59.

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Table 61

BACKSTREAMING MEASUREMENTS BY ULTRAVIOLET ABSORBANCE AT STATION 7

Run	Bakeout Temp., °F	Run, hr	Trap[a] Temp., °F	System Pressure, torr — Room Temp.	System Pressure, torr — LN_2 Temp.	Deposit Weight, mg/cm² — Total	Deposit Weight, mg/cm² — Analytical Blank[c]	Net Backstreaming — Deposit, mg/cm²	Net Backstreaming — Rate, mg/cm²-min
189	RT	290	RT	1×10^{-8}	7×10^{-9}	99.0×10^{-4}	21.9×10^{-4}	77.1×10^{-4}	0.446×10^{-6} [b]
194	RT	72	-75	6×10^{-9}	5×10^{-9}	16.2×10^{-4}	16.2×10^{-4}	0	0
199	RT	292	-75	2×10^{-9}	2×10^{-10}	20.4×10^{-4}	8.8×10^{-4}	11.6×10^{-4}	0.066×10^{-6}
201	212	148	-75	2×10^{-9}	2×10^{-10}	38.6×10^{-4}	5.3×10^{-4}	33.3×10^{-4}	0.376×10^{-6}
							40.4×10^{-4} [e]	-1.8×10^{-4}	-0.020×10^{-6}
206	212	552	-75/-44[d]	4×10^{-9}	4×10^{-9}	12.3×10^{-4}	-1.8×10^{-4} [e]	14.1×10^{-4}	0.043×10^{-6}
							-3.6×10^{-4}	15.9×10^{-4}	0.048×10^{-6}
215	212	149	-21/10[d]	1×10^{-9}	2×10^{-10}	13.3×10^{-4}	7.3×10^{-4}	6.0×10^{-4}	0.067×10^{-6}
							37.5×10^{-4}	-24.2×10^{-4}	-0.27×10^{-6}
221	212	315	-75	3×10^{-9}	2×10^{-10}	7.4×10^{-4}	4.2×10^{-4}	3.2×10^{-4}	0.039×10^{-6}
							7.0×10^{-4}	0.4×10^{-4}	0.002×10^{-6}
241	212	1036	-75	6×10^{-10}	1×10^{-10}	22.8×10^{-4}	1.7×10^{-4}	21.1×10^{-4}	0.037×10^{-6}
							8.8×10^{-4}	14.8×10^{-4}	0.023×10^{-6}
							2.8×10^{-4}	20.0×10^{-4}	0.032×10^{-6}
							2.8×10^{-4}	20.0×10^{-4}	0.032×10^{-6}

[a] Trap temperature refers to plate temperature in the turbomolecular pumps.

[b] Plate was not cooled.

[c] Successive analytical blanks each preceded by 3 isooctane washes.

[d] Refrigerator not cooling satisfactorily.

[e] Negative value found due to changes in isooctane due to standing.

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Table 62

BACKSTREAMING MEASUREMENTS BY ULTRAVIOLET ABSORBANCE AT STATION 8

Run	Bakeout Temp., °F	Run, hr	Trap Temp.,[a] °F	System Pressure, torr — Room Temp.	System Pressure, torr — LN_2 Temp.	Deposit Weight, mg/cm² — Total	Deposit Weight, mg/cm² — Analytical Blank[b]	Net Backstreaming — Deposit, mg/cm²	Net Backstreaming — Rate, mg/cm²-min
198	RT	391	-75	7×10^{-9}	8×10^{-10}	30.2×10^{-4}	11.2×10^{-4}	19.0×10^{-4}	0.081×10^{-6}
							10.5×10^{-4}	19.7×10^{-4}	0.084×10^{-6}
200	212	148	-77	8×10^{-9}	6×10^{-10}	12.6×10^{-4}	5.2×10^{-4}	7.4×10^{-4}	0.082×10^{-6}
							4.2×10^{-4}	8.4×10^{-4}	0.094×10^{-6}
209	212	809	-75/-40[c]	3×10^{-9}	4×10^{-10}	-4.6×10^{-4}[d]	-4.6×10^{-4}[e]	0	0
214	212	149	-75	9×10^{-9}	1×10^{-9}	12.4×10^{-4}	1.8×10^{-4}	-6.4×10^{-4}	-0.013×10^{-6}
							8.9×10^{-4}	2.5×10^{-4}	0.028×10^{-6}
220	212	514	-75	7×10^{-9}	2×10^{-9}	15.8×10^{-4}	23.0×10^{-4}	-10.4×10^{-4}	-0.117×10^{-6}
							6.3×10^{-4}	9.5×10^{-4}	0.031×10^{-6}
							7.0×10^{-4}	8.8×10^{-4}	0.029×10^{-6}
Added Chevron baffle									
242[f]	212	1052	RT[g]	2×10^{-8}	2×10^{-9}	264.0×10^{-4}	9.1×10^{-4}	254.9×10^{-4}	0.402×10^{-6}
							7.3×10^{-4}	256.7×10^{-4}	0.405×10^{-6}
							8.8×10^{-4}	255.2×10^{-4}	0.403×10^{-6}
							12.3×10^{-4}	251.7×10^{-4}	0.398×10^{-6}

[a] Trap temperature refers to plate temperature in the turbomolecular pumps.

[b] Successive analytical blanks, each preceded by 3 isooctane washes.

[c] Refrigerator not cooling satisfactorily.

[d] Negative value found for original sample as well as analytical blank.

[e] Negative value found due to changes in isooctane on standing.

[f] Vacuum system of test head, baffle and turbomolecular pump inlet baked at 100°C for 18 hr.

[g] Trap and plate not cooled.

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Table 63

STANDARDIZATION VALUES FOR ULTRAVIOLET ABSORBANCE VERSUS GAS CHROMATOGRAPHY

Run	Peak Height, cm C_{16}	Peak Height, cm C_{17}	Reliability Ratio	Calculated Amounts C_{16}	Calculated Amounts C_{17}	Weight Average, μg	Wt. Value By UV, μg	Percent Ratio GC/UV
Standard	45.9 (46.3) 46.7	40.8 (40.3) 39.8	1.17	40.2	40.2	40.2	40.2	
189	22.7	22.1	1.03	19.7	22.3	21.0	146	14.4%
189A	1.1	1.2	0.92	0.9	1.2	1.0	64.8	1.5%
198	4.7	4.5	1.0	4.1	4.5	4.3	88	4.8%
201	7.4	5.9	1.25	6.4	6.0	6.2	113.6	5.5%
201B	5.5	4.4	1.25	4.8	4.4	4.6	118.4	3.9%
214	2.0	2.0	1.0	1.7	2.02	2.0	90.5	2.2%
214B	2.6	6.8	0.38	2.3	6.9	4.6	100.5	4.6%
215	8.5	7.9	1.73	7.4	4.9	6.2	98.0	6.3%
215A	2.4	2.6	0.92	2.1	2.6	2.4	109.4	2.2%

Table 64

COMPARISON OF RUN DATA ON ULTRAVIOLET ABSORBANCE VERSUS GAS CHROMATOGRAPHY

Run	UV Absorbance		Gas Chromatograph		Backstreaming Net Deposit, mg/cm^2	
	Total Weight, mg/cm^2	Analytical Blank, mg/cm^2	Total Weight, mg/cm^2	Analytical Blank, mg/cm^2	UV	GC
189	99.0×10^{-4}	21.9×10^{-4}	14.3×10^{-4}	0.34×10^{-4}	77.1×10^{-4}	14.0×10^{-4}
201	38.6×10^{-4}	40.4×10^{-4}	2.1×10^{-4}	1.6×10^{-4}	-1.8×10^{-4}	0.5×10^{-4}
214	12.3×10^{-4}	22.8×10^{-4}	0.27×10^{-4}	1.04×10^{-4}	-10.5×10^{-4}	-0.77×10^{-4}
215	13.3×10^{-4}	37.2×10^{-4}	0.84×10^{-4}	0.82×10^{-4}	-23.9×10^{-4}	0.02×10^{-4}

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IITRI-C6030-10
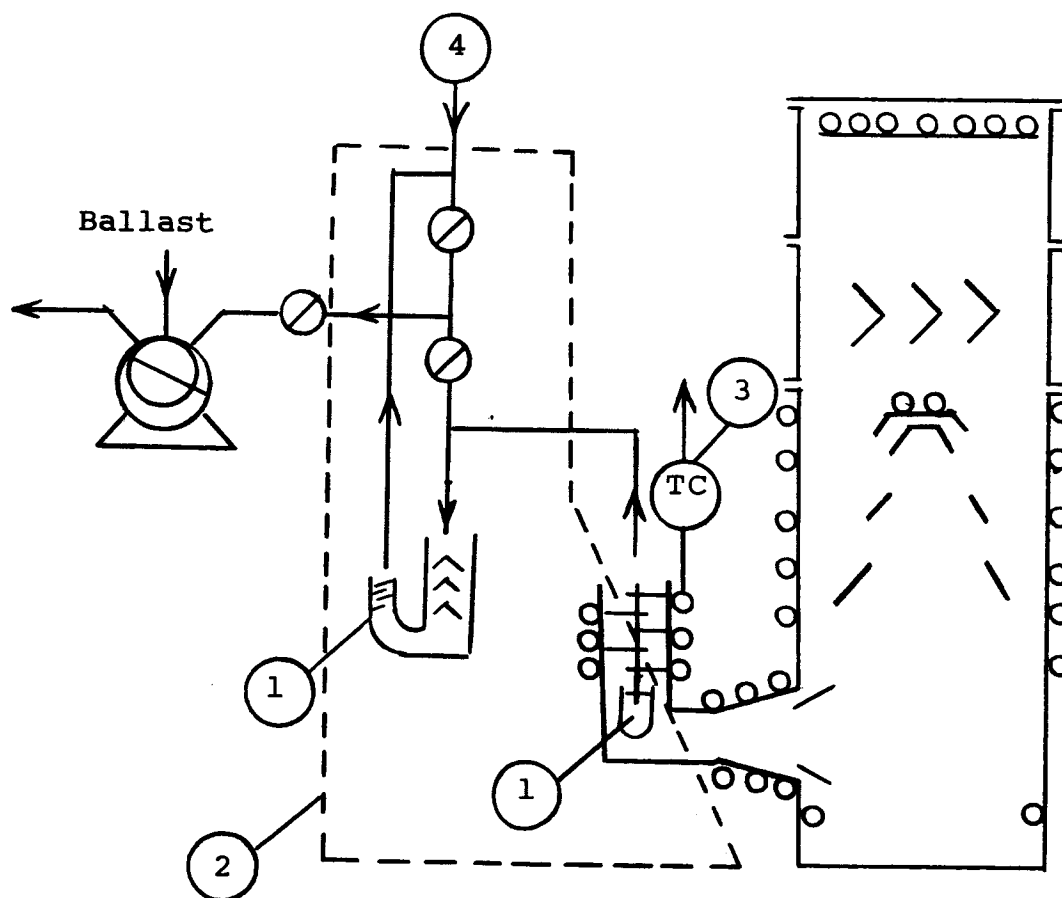
1   Hold-up alembic

2   Dotted section Station 3 only

3   Exit temperature control on Station 1 only

4   Air leak, guard vacuum, quench coils

     See Table 13 for schedule of pump, baffle and oil

Figure 1

SCHEMATIC OF ARRANGEMENT OF TEST STATIONS

IITRI-C6030-10
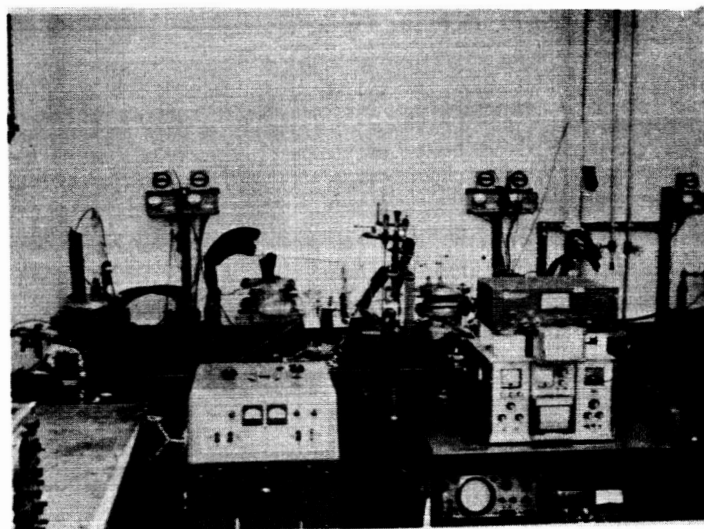
Figure 2

PHOTOGRAPH OF TEST FACILITY
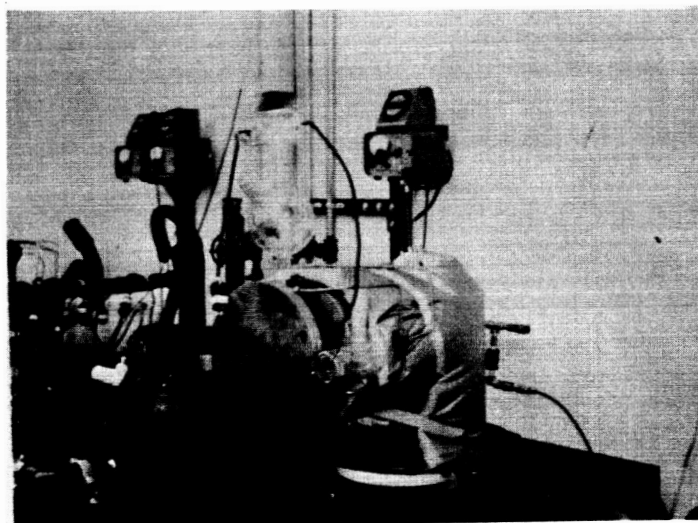
Figure 3

PHOTOGRAPH OF A RIGHT-ANGLE ELBOW
(Station 5)

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IITRI-C6030-10
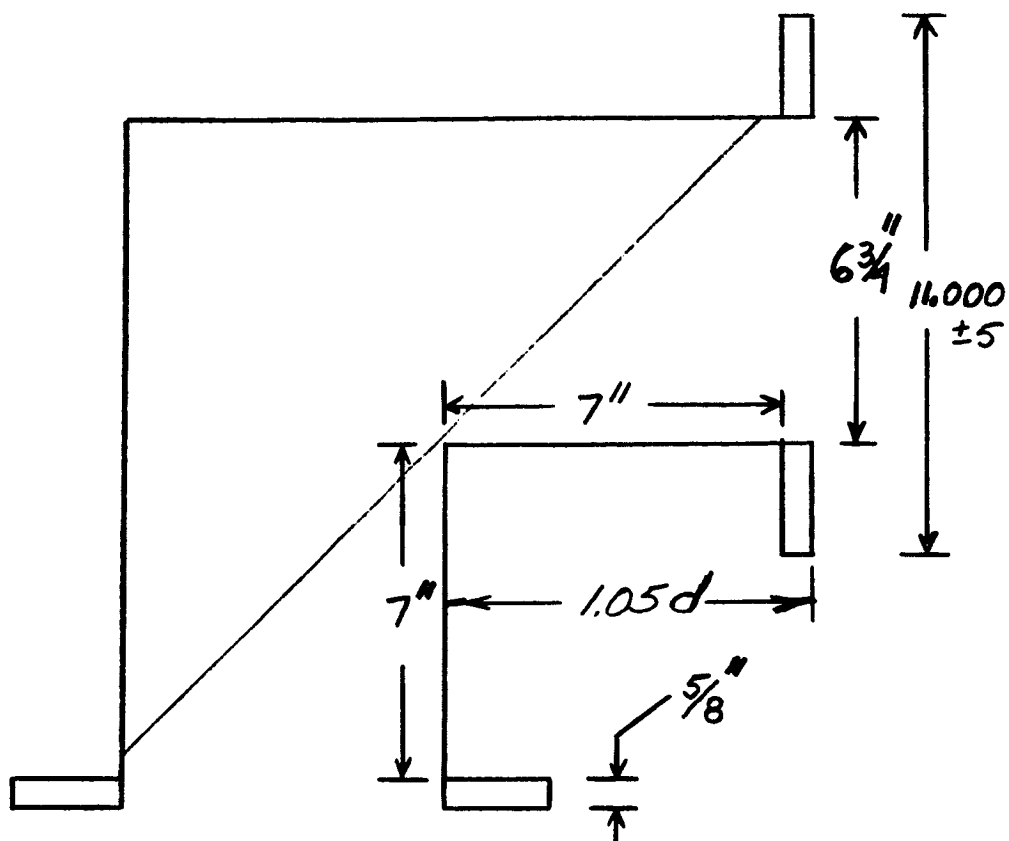
Figure 4

SCHEMATIC OF RIGHT-ANGLE ELBOW BAFFLE
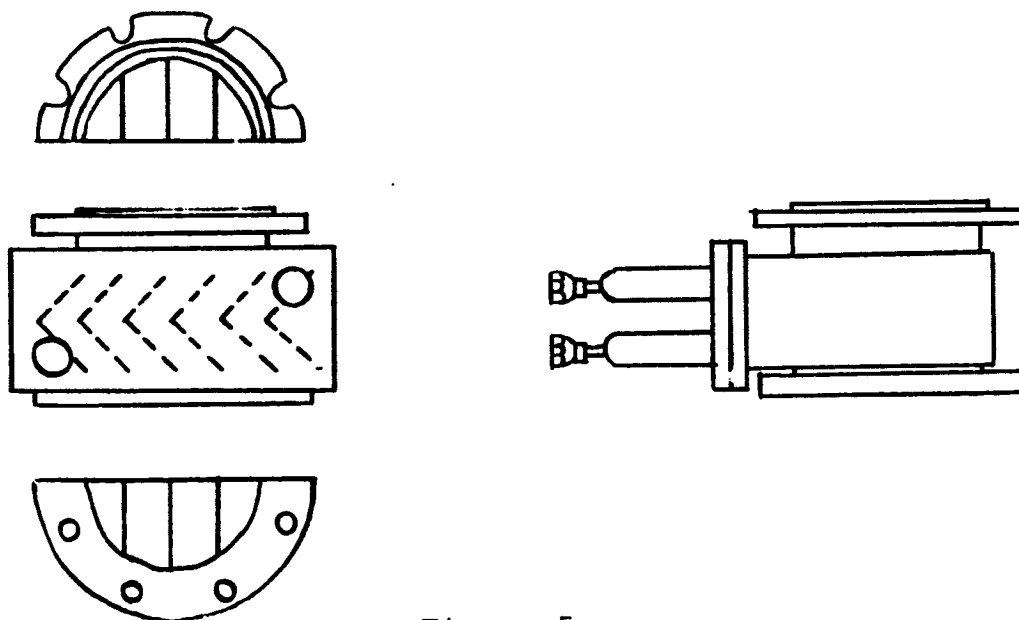
Figure 5

CVC– BC–61 CHEVRON BAFFLE

134
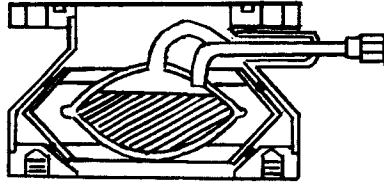
Figure 6

NRC-HN-6 CIRCULAR CHEVRON BAFFLE
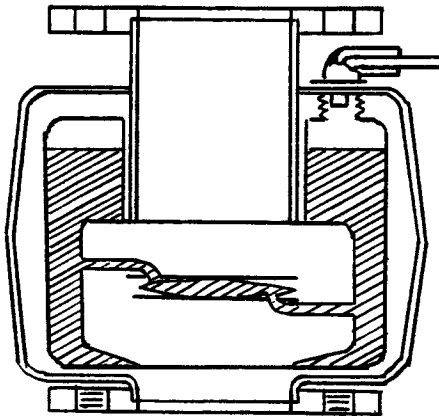
Figure 7

GRANVILLE PHILLIPS SERIES 251 CRYOSORB BAFFLE

135
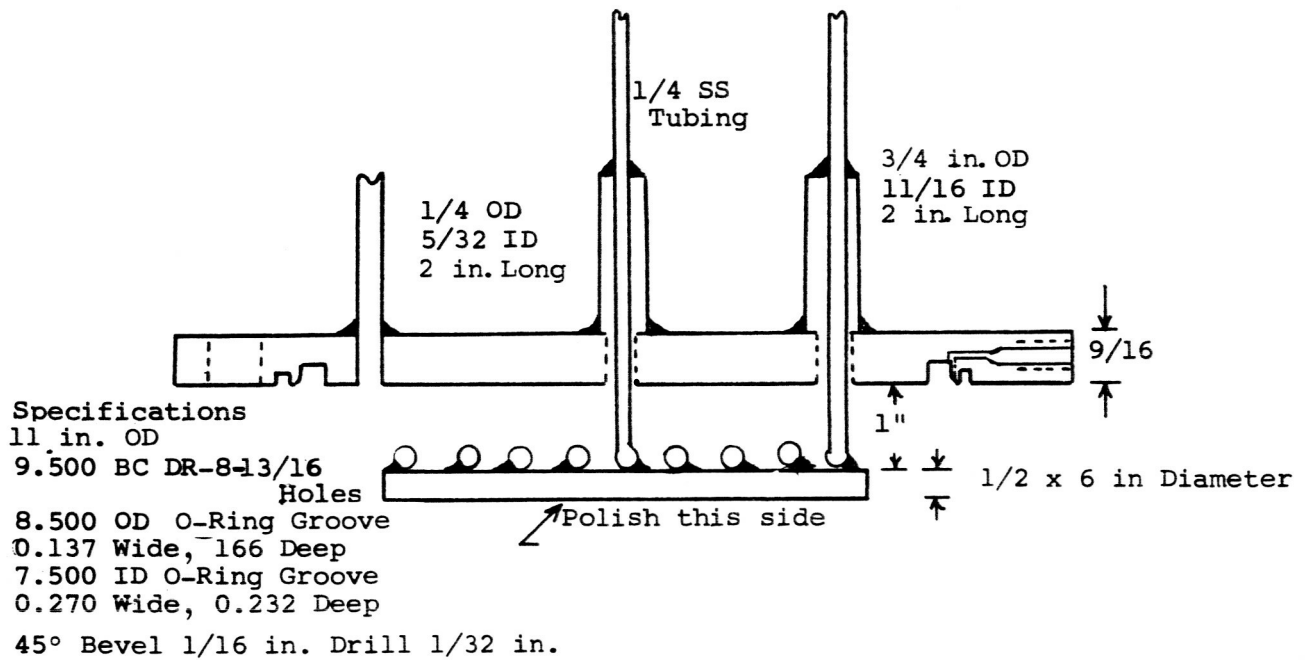
Figure 8

COLLECTION PLATE DETAILS
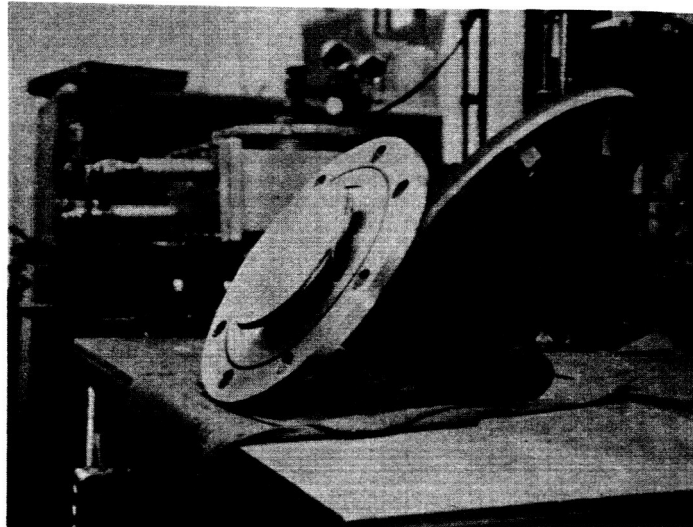
Figure 9

PHOTOGRAPH OF A COLLECTION PLATE

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IITRI-C6030-10
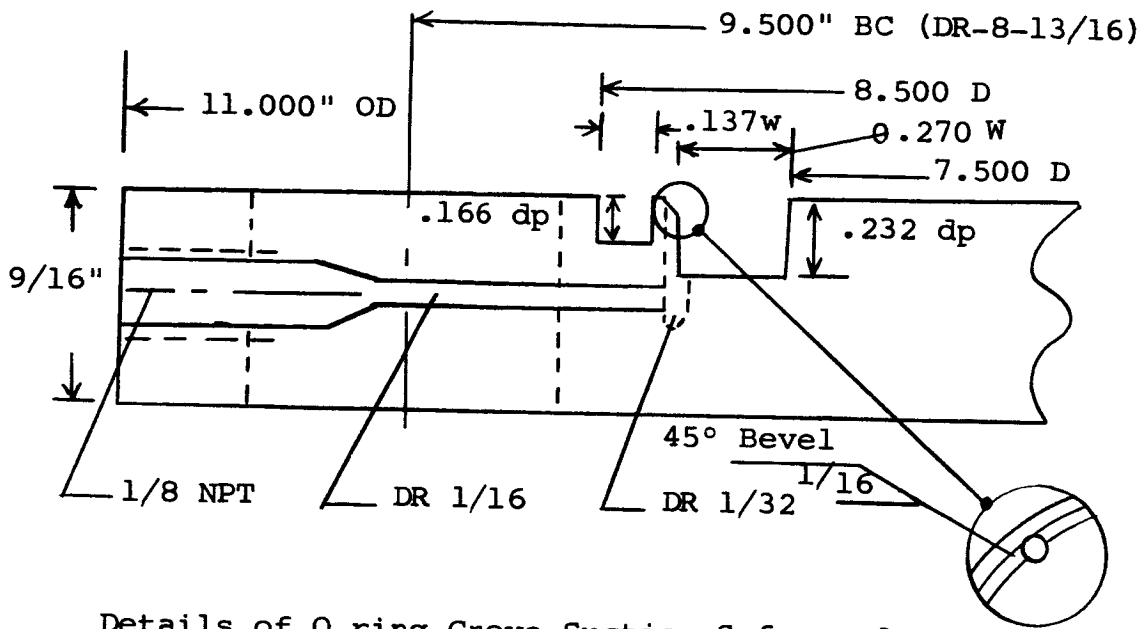
Details of O-ring Grove Suction Safeguard
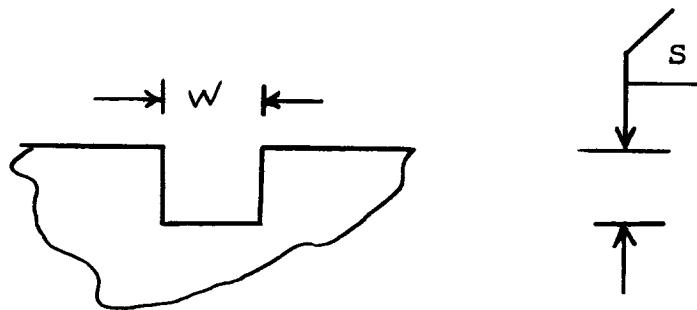
dn – nominal diameter of O-ring
da – actual diameter of O-ring
S = dn – (0.009) $\left[dn / (1/8) \right]$
W = π/4 (da)^2/0.97 S

Figure 10

SPECIFICATIONS OF O-RING GROOVE
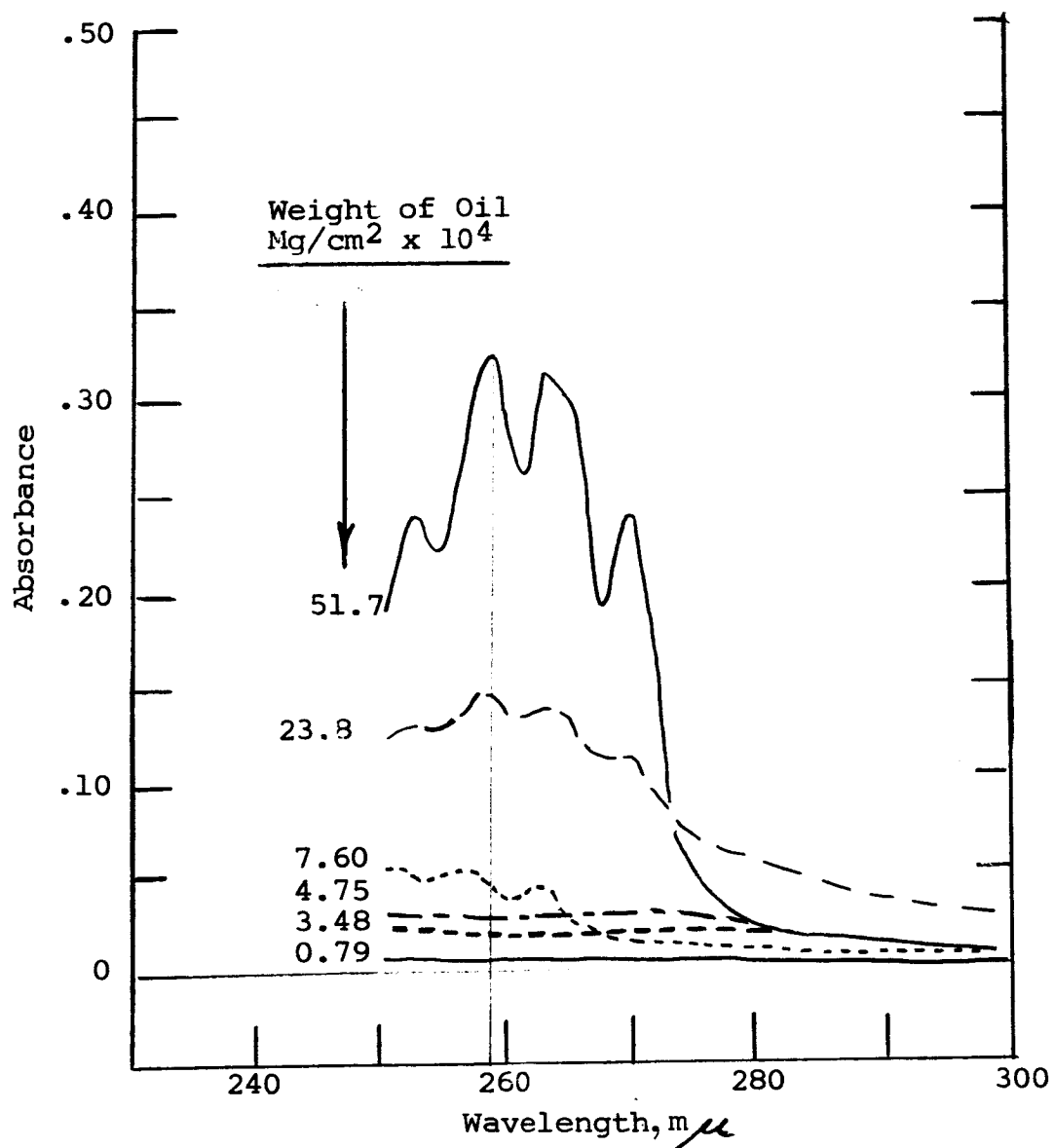
Figure 11

ULTRAVIOLET ABSORBANCE CURVES FOR SMALL
AMOUNTS OF DC 705

138
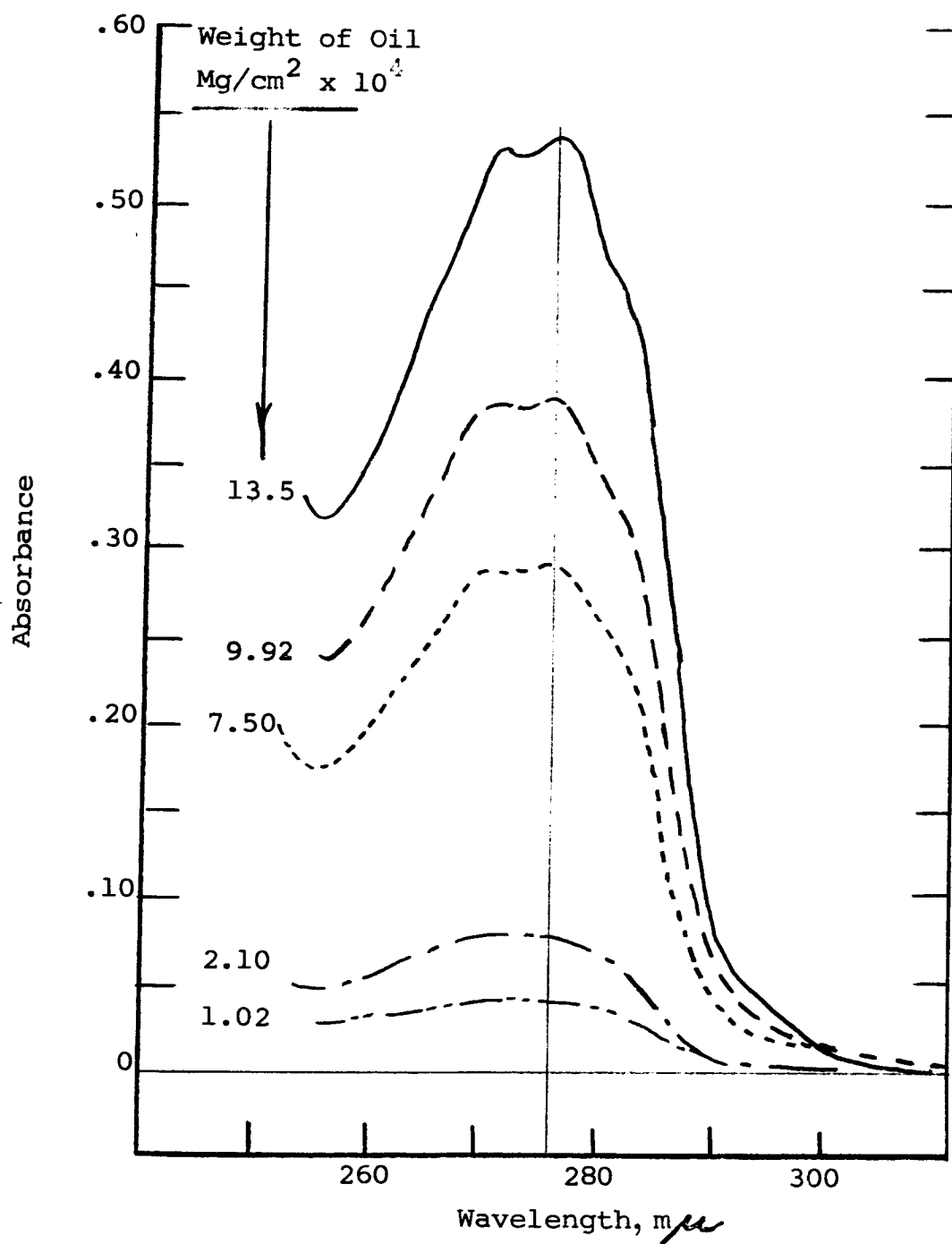
Figure 12

ULTRAVIOLET ABSORBANCE CURVES FOR
SMALL AMOUNTS OF PENTAPHENYL ETHERS

IITRI-C6030-10

Loenco Dual-Flame
Gas Chromatograph 3 ft × 3/16 in.
3% SE 30 Column
Attenuation = 8X
Detention θ DC 705 = 5.9 cm
Sensitivity = 1.34 cm²/μg
Helium pressure 10 psig
T = 300°C

$A = 3.78 \text{ cm}^2$
$S = \dfrac{3.78}{2.62} = 1.44 \dfrac{\text{cm}^2}{\mu g}$

θ=5.8 cm

$A = 1.12 \text{ cm}^2$
$S = \dfrac{1.12}{1.04} = 1.07 \dfrac{\text{cm}^2}{\mu g}$

θ=5.9 cm

$A = .610 \text{ cm}^2$
$S = \dfrac{.610}{.523} = 1.17 \dfrac{\text{cm}^2}{\mu g}$

θ=6.0 cm
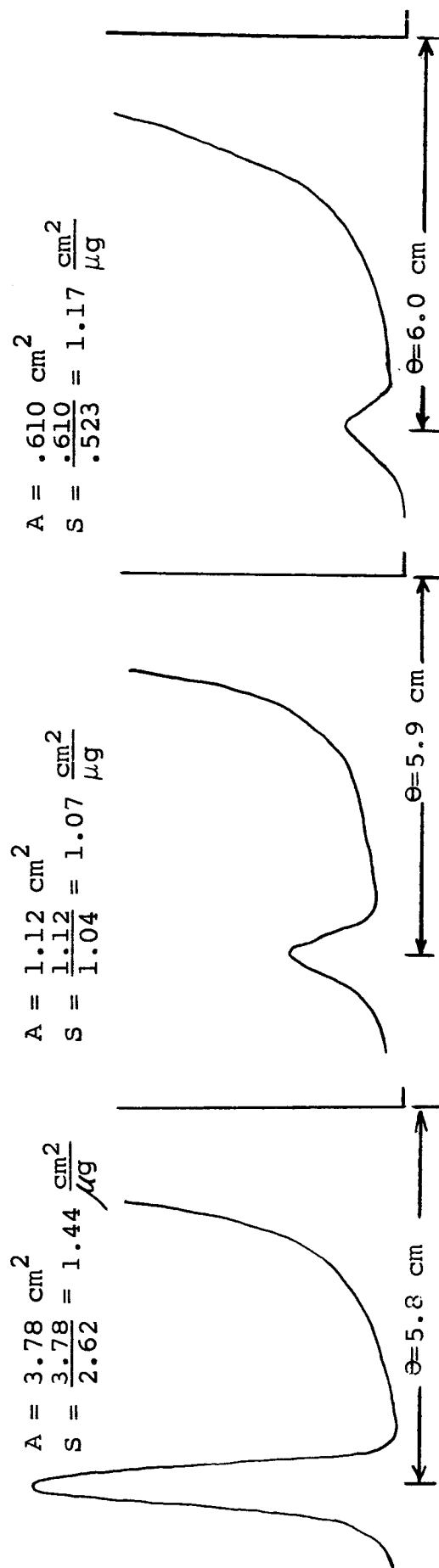
Figure 13

CALIBRATION CURVE FOR DC 705

IITRI-C6030-10

Loenco Dual-Flame
Gas Chromatograph
3% SE 30 Column 6 ft x 3/16 in.
Attenuation = 8X
Detention θ DC 704 = 2.7 cm
Sensitivity = 1.39 cm²/μg
Helium pressure 10 psig
T = 300°C

$A = 3.94 \ cm^2$
$S = \dfrac{3.94}{2.54} = 1.56 \ \dfrac{cm^2}{\mu g}$

$A = 1.35 \ cm^2$
$S = \dfrac{1.35}{1.01} = 1.33 \ \dfrac{cm^2}{\mu g}$

$A = .542 \ cm^2$
$S = \dfrac{.542}{.508} = 1.07 \ \dfrac{cm^2}{\mu g}$

DC 704
2.54 μg

DC 704
1.01 μg

DC 704
0.508 μg

θ=2.7 cm

θ=2.7 cm

θ=2.8 cm
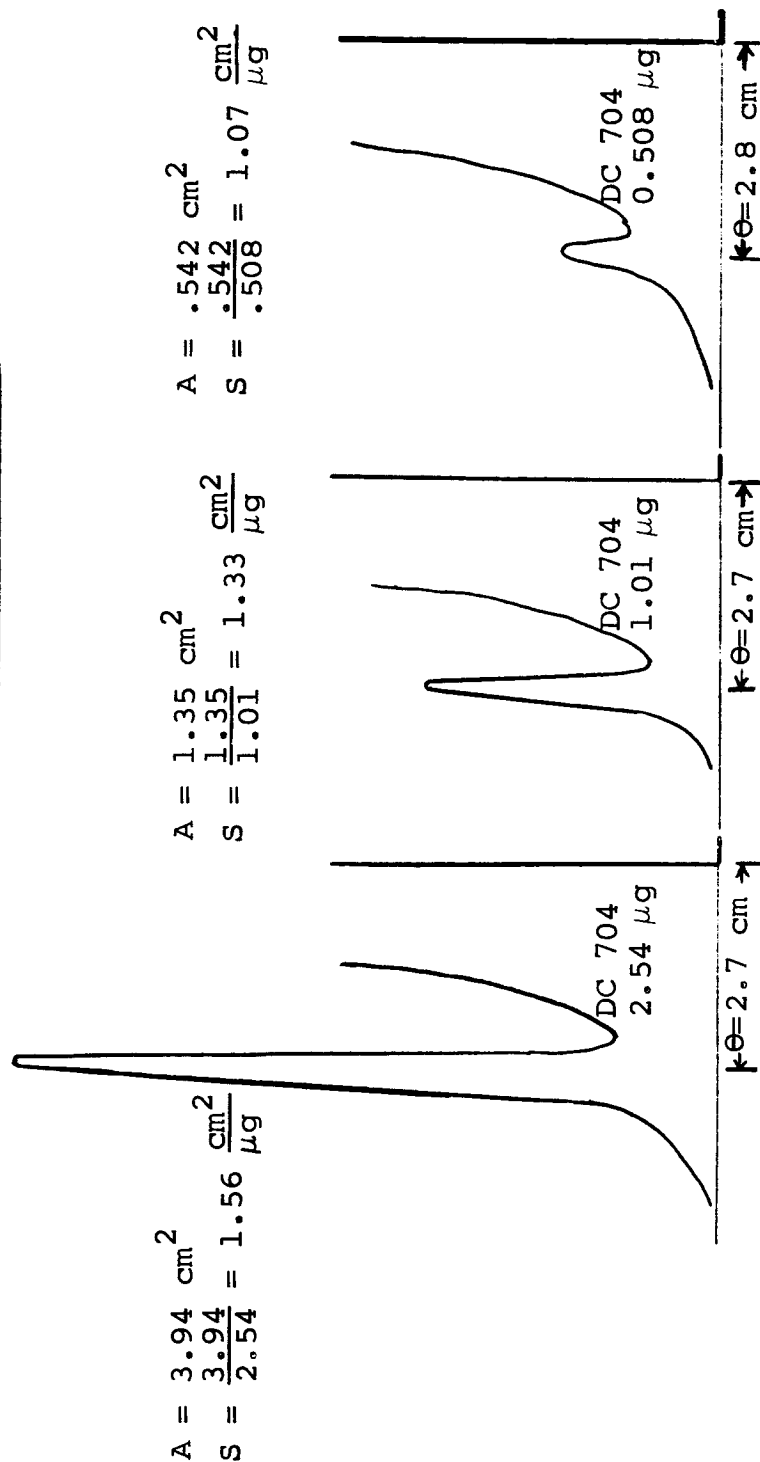
Figure 14

CALIBRATION CURVE FOR DC 704

141

Loenco Dual-Flame
Gas Chromatograph
5% SE 30 Column 6 ft x 3/16 in.
Attenuation = 4, 8, 32
Detention @ DC 704 = 6.8 cm
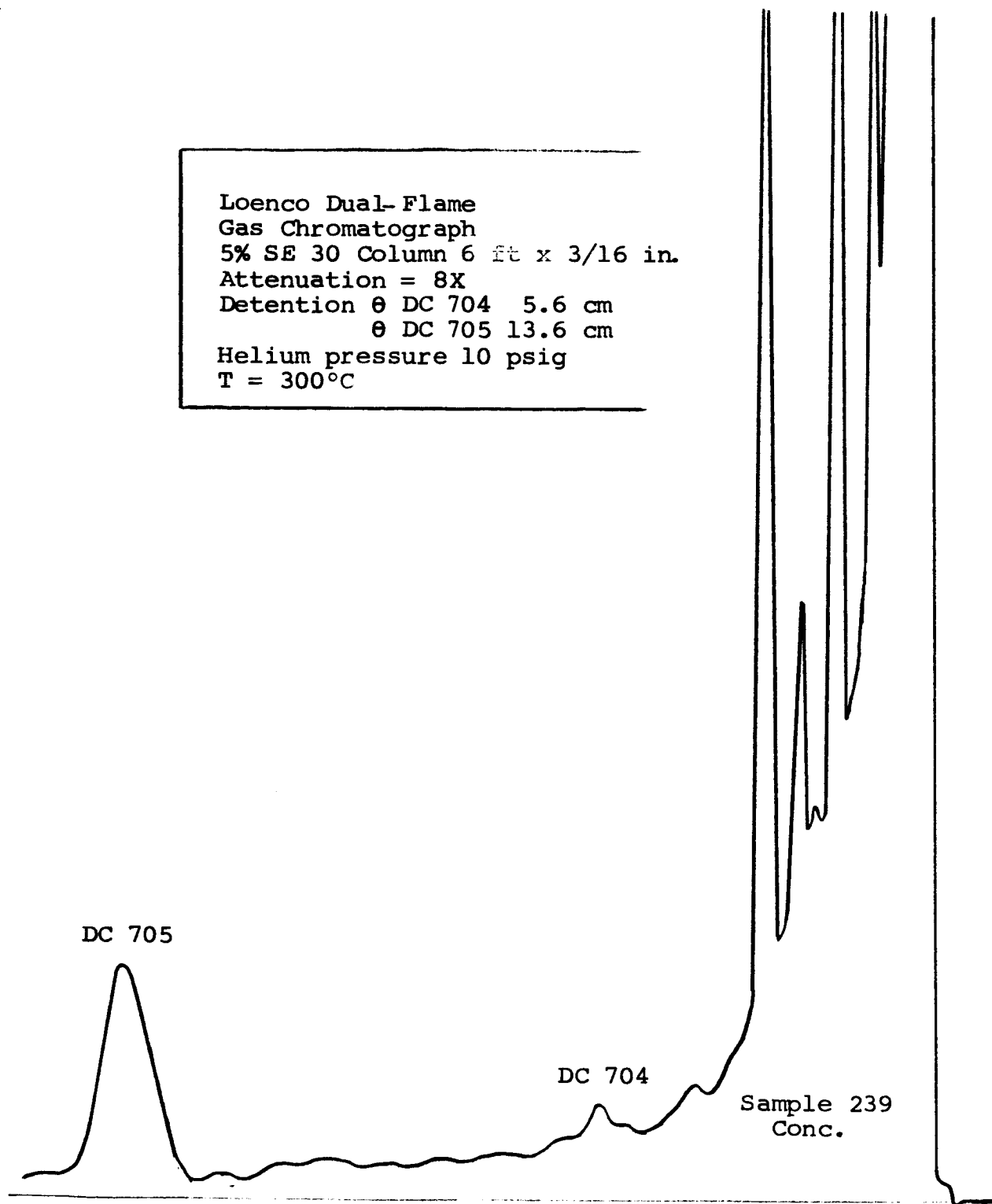DC 705 = 16.0 cm

Helium Pressure 40 psig
T = 300°C

0.91%
DC 704

4X

A = 0.11 cm² CORR.

32X

A = 9.25 cm² CORR.

TOTAL AREA = 9.36 cm²
DC 704 / TOTAL AREA = 0.012

2.4%
DC 704

8X

A = 0.27 cm² CORR.

A = 9.25 cm² CORR.

32X

A = 9.25 cm² CORR.

TOTAL AREA = 9.49 cm²
DC 704 / TOTAL AREA = 0.025
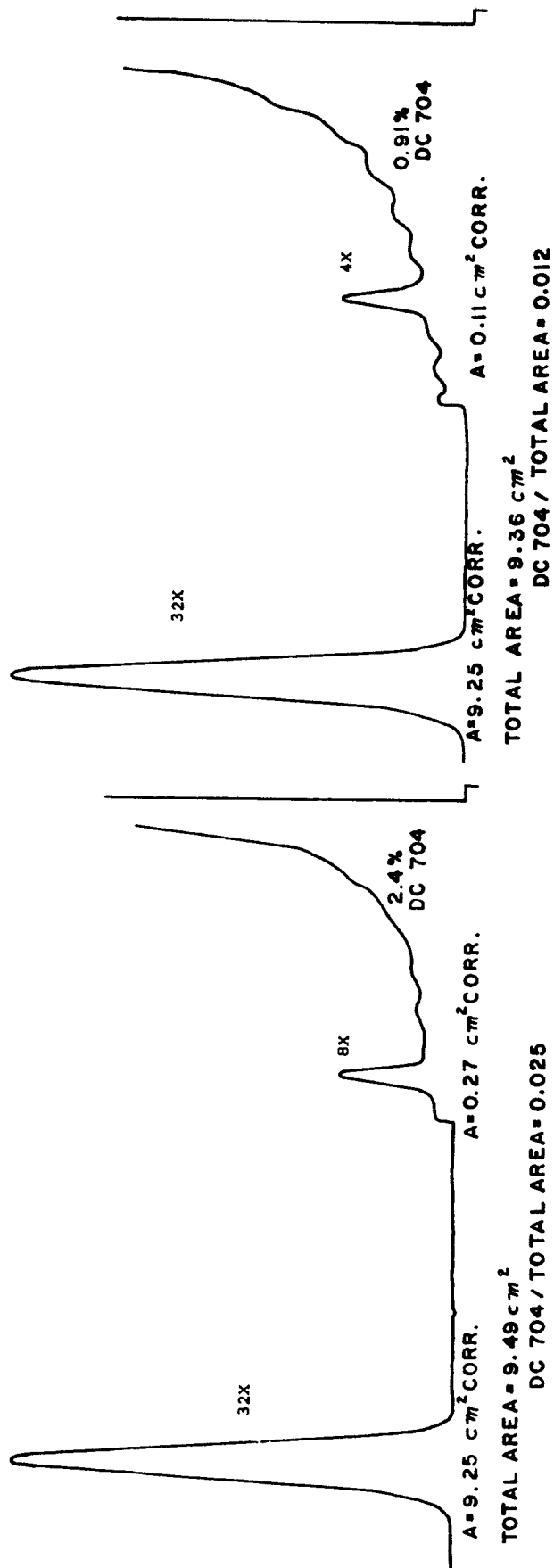
FIGURE 15

CALIBRATION CURVES FOR MIXTURES OF DC 705 / DC 704

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Loenco Dual-Flame
Gas Chromatograph
5% SE 30 6 ft x 3/16 in
Attenuation = 32X
Detention θ DC 704   7.1 cm
           DC 705 17.1 cm
Helium Pressure 40 psig
T = 300°C

33.3 %
DC 704

A=4.85cm²CORR.

TOTAL AREA=14.10cm²

DC 704 / TOTAL AREA = 0.344

A=9.25cm²CORR.

50 %
DC 704

A=9.3cm²CORR.

TOTAL AREA=0.50

DC 704 / TOTAL AREA = 0.50

A=9.25cm²CORR.

TOTAL AREA=18·55 cm²

DC 704 / TOTAL AREA = 0.50
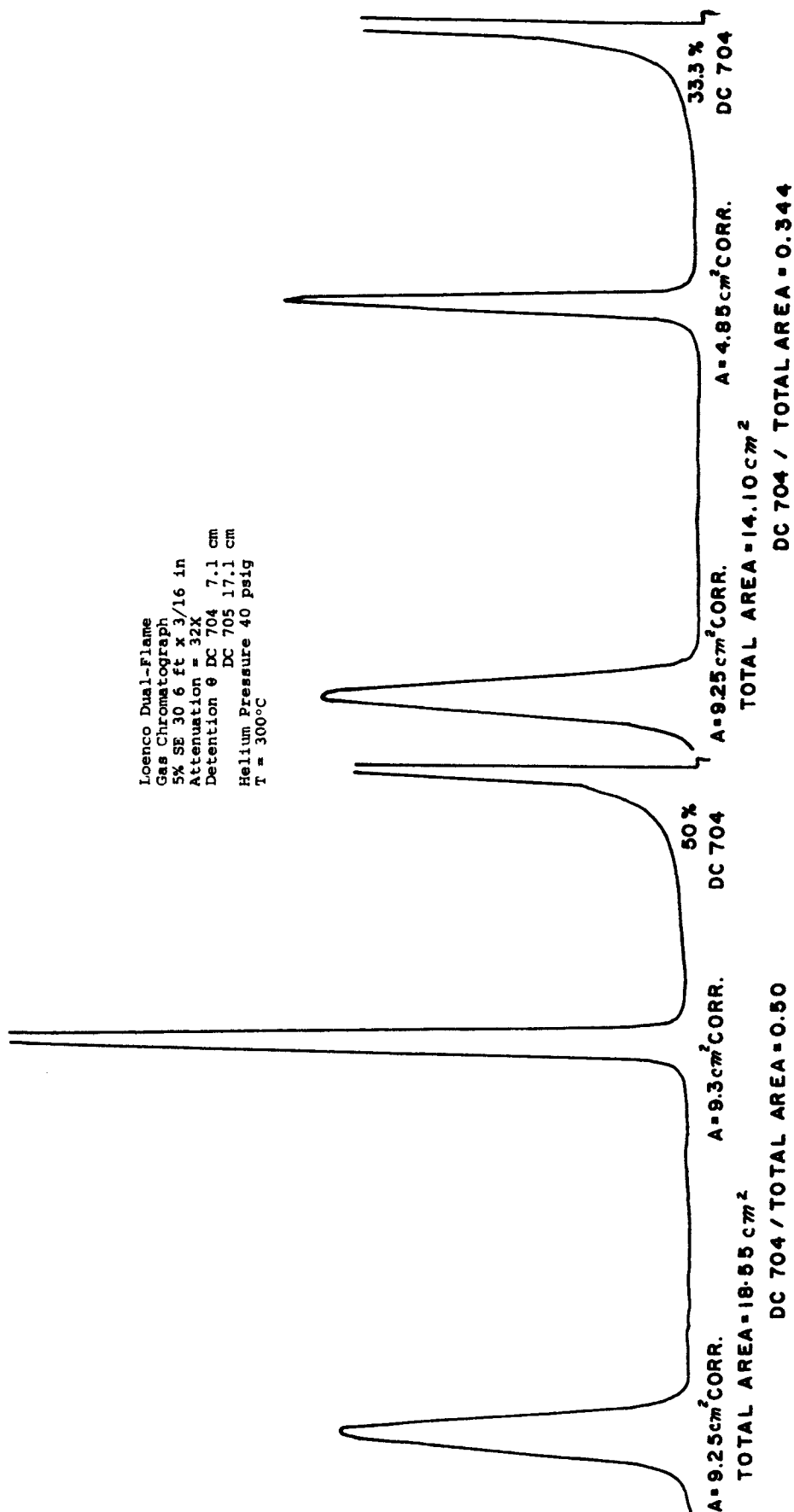
FIGURE 15 (CONT.)

CALIBRATION CURVES FOR MIXTURES OF DC 705 / DC 704

Loenco Dual-Flame
Gas Chromatograph
5% SE 30 Column 6 ft x 3/16 in
Attenuation = 32X
Detention DC 704 = 6.8 cm
Detention DC 705 = 16.5 cm
Helium pressure 40 psig
T = 300°C

9.1%
DC 704

A=1.12 cm² CORR.

A=9.25 cm² CORR.
TOTAL AREA = 10.37 cm²
DC 704 / TOTAL AREA = 0.109

20%
DC 704

A=2.21cm² CORR.
DC 704 / TOTAL AREA = 0.194

A=9.25cm² CORR.
TOTAL AREA = 11.47 cm²
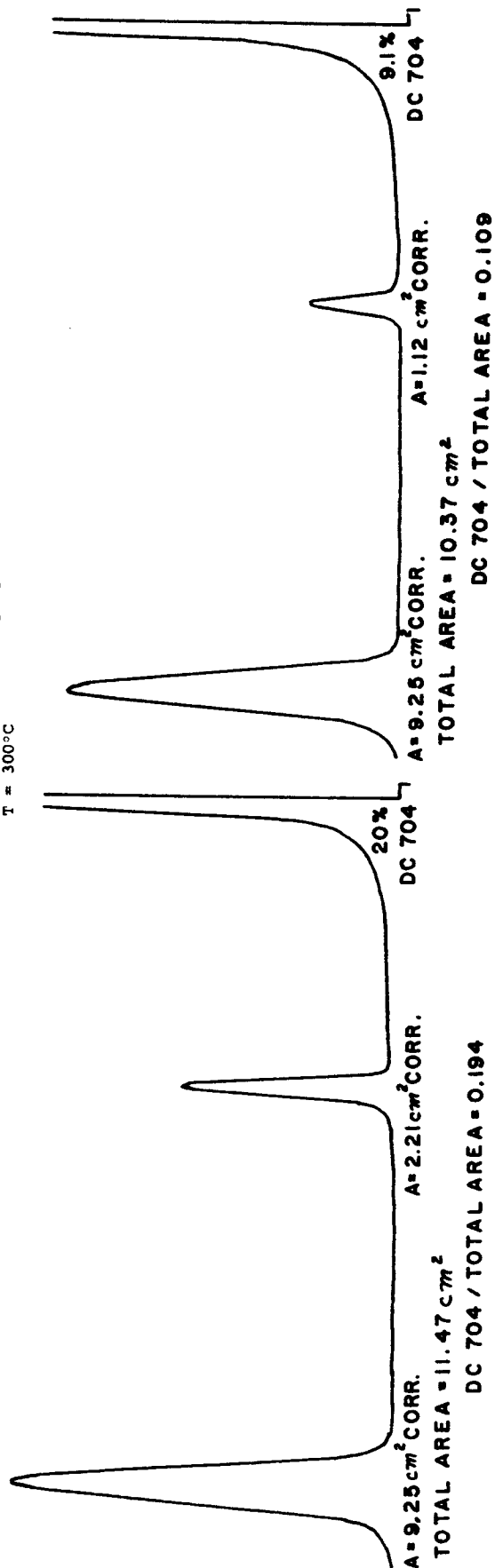
FIGURE 15 (CONT.)

CALIBRATION CURVES FOR MIXTURES OF DC 705 / DC 704
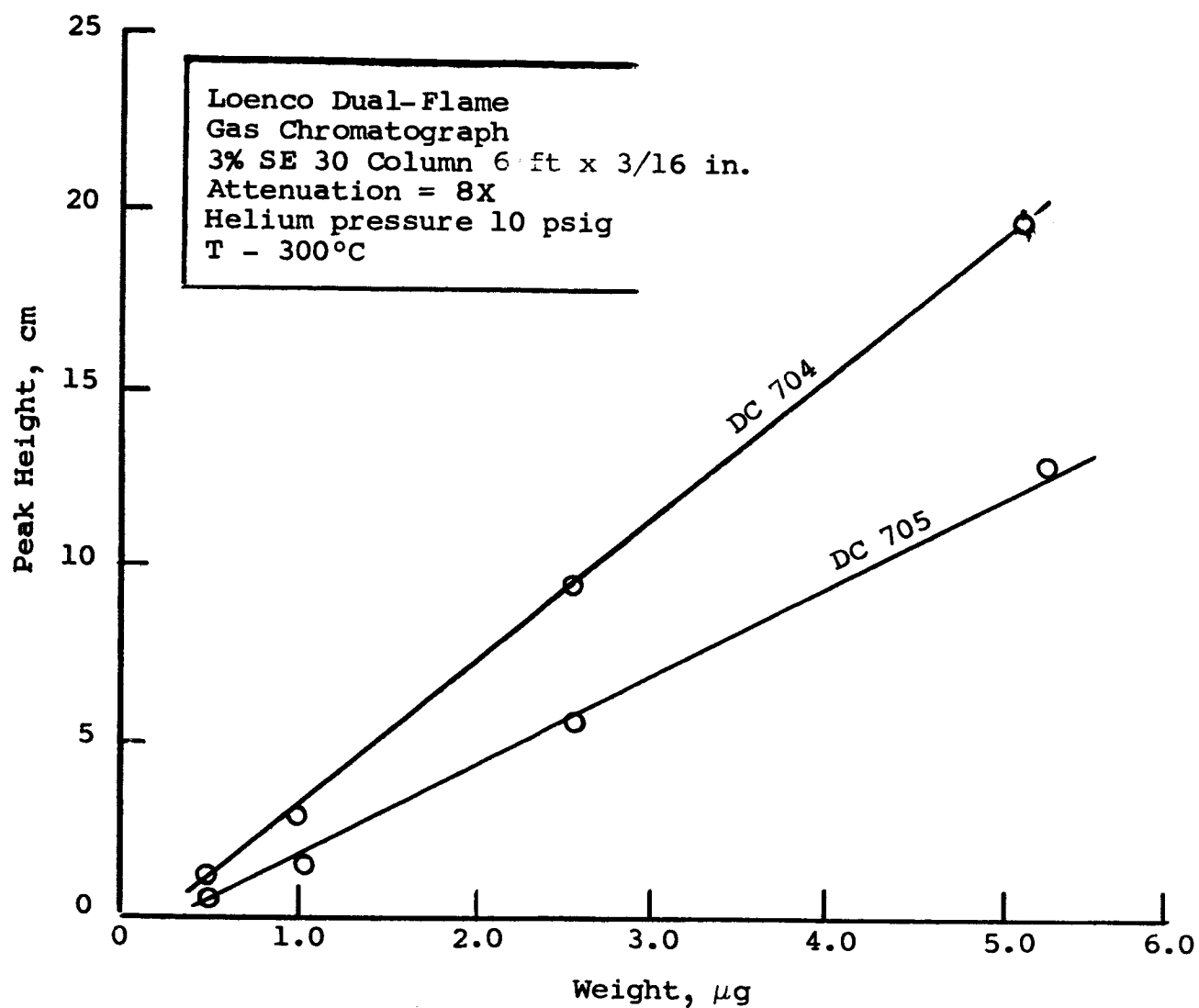
Figure 16

SENSITIVITY IN PEAK HEIGHT VERSUS WEIGHT FOR DC 705 AND DC 704

145
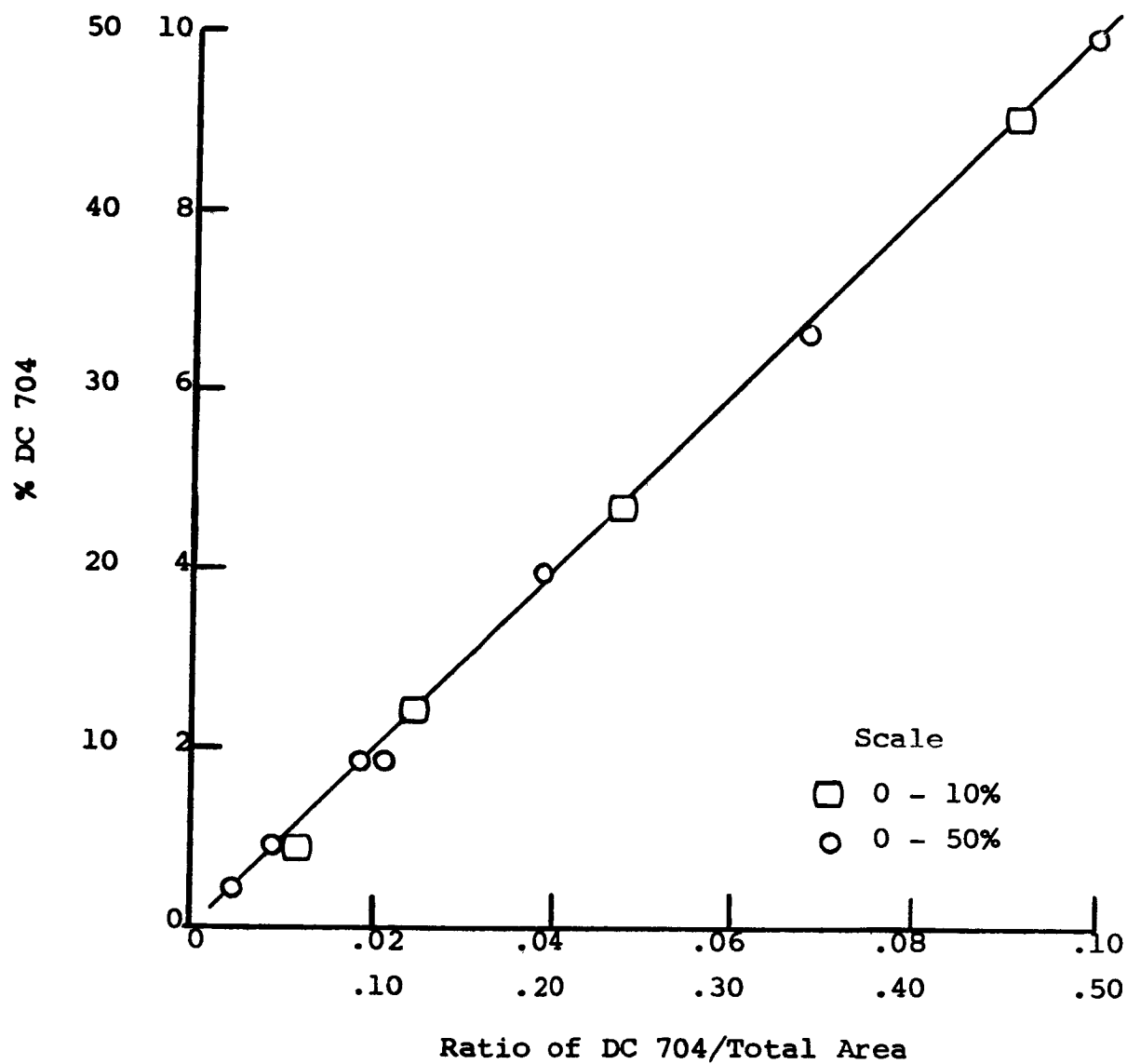
Figure 17

PERCENT OF DC 704 IN DC 705

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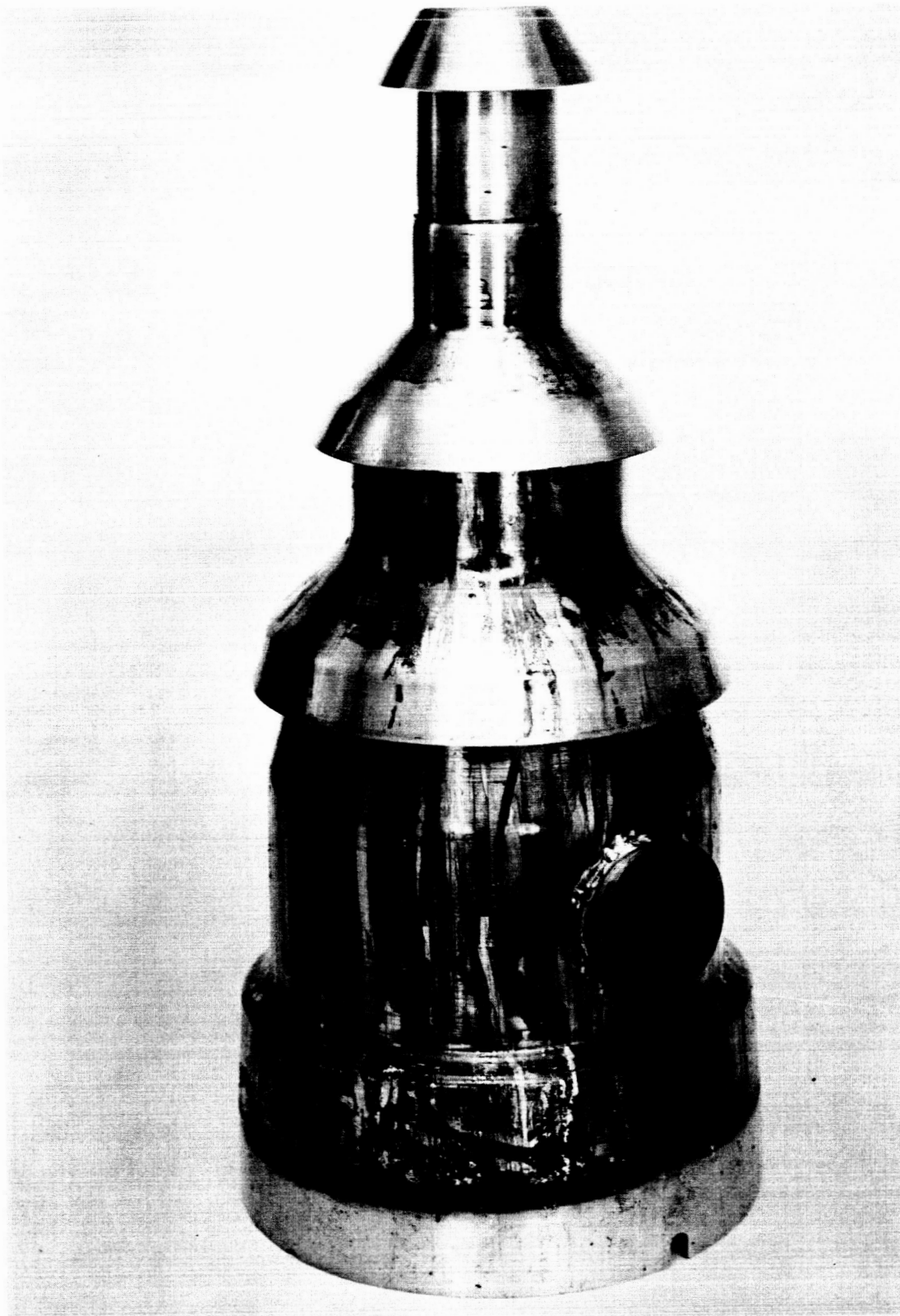
Figure 18

EXTERNAL JET SURFACES OF STATION 5

AFTER 1 YEAR OF EXPOSURE TO CONVALEX 10

147
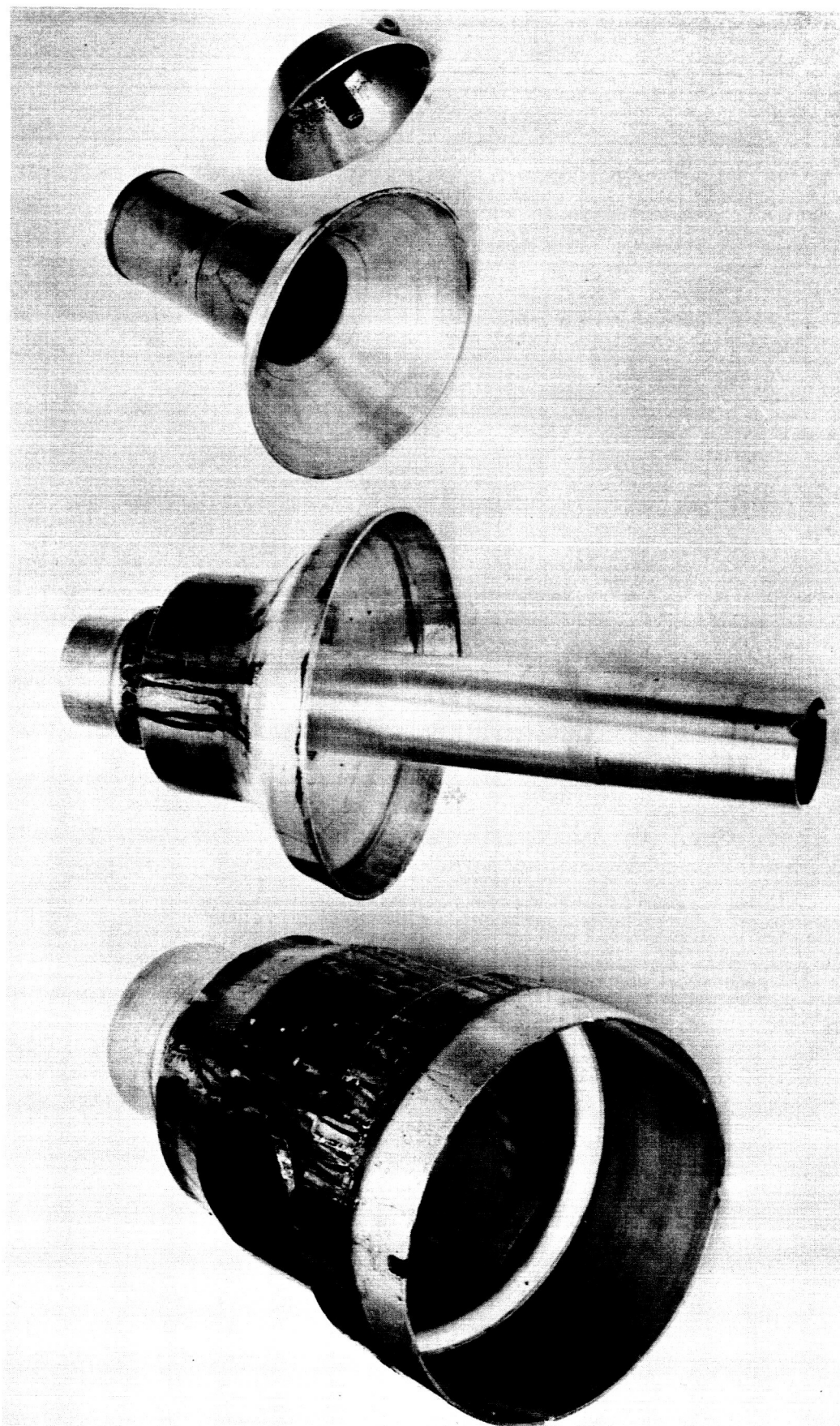
Figure 19

INNER AND OUTER JET SURFACES OF STATION 5

AFTER 1 YEAR OF EXPOSURE TO CONVALEX 10

IITRI-C6030-10
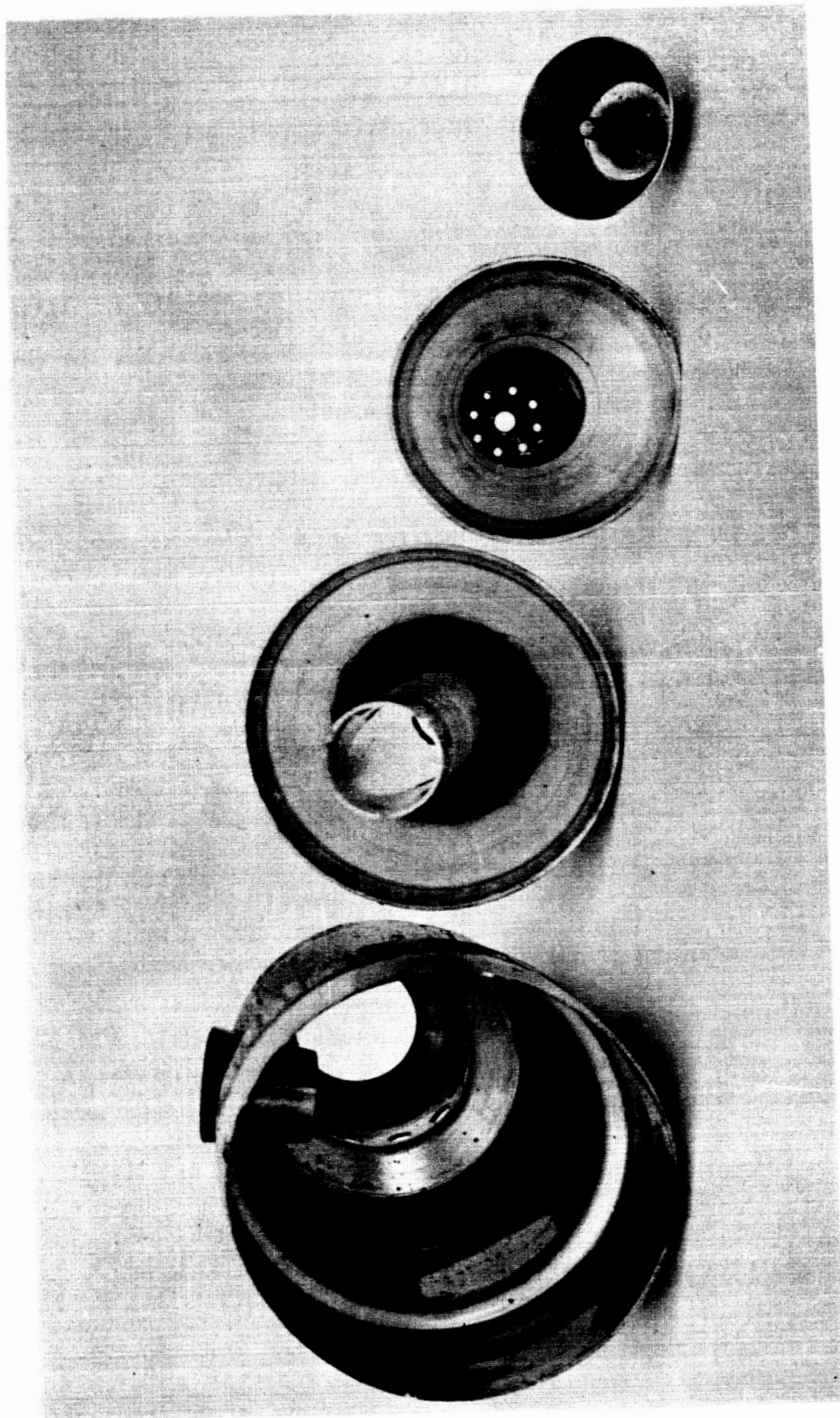
Figure 20

INNER JET SURFACES OF STATION 5

AFTER 1 YEAR OF EXPOSURE TO CONVALEX 10

149

IITRI-C6030-10
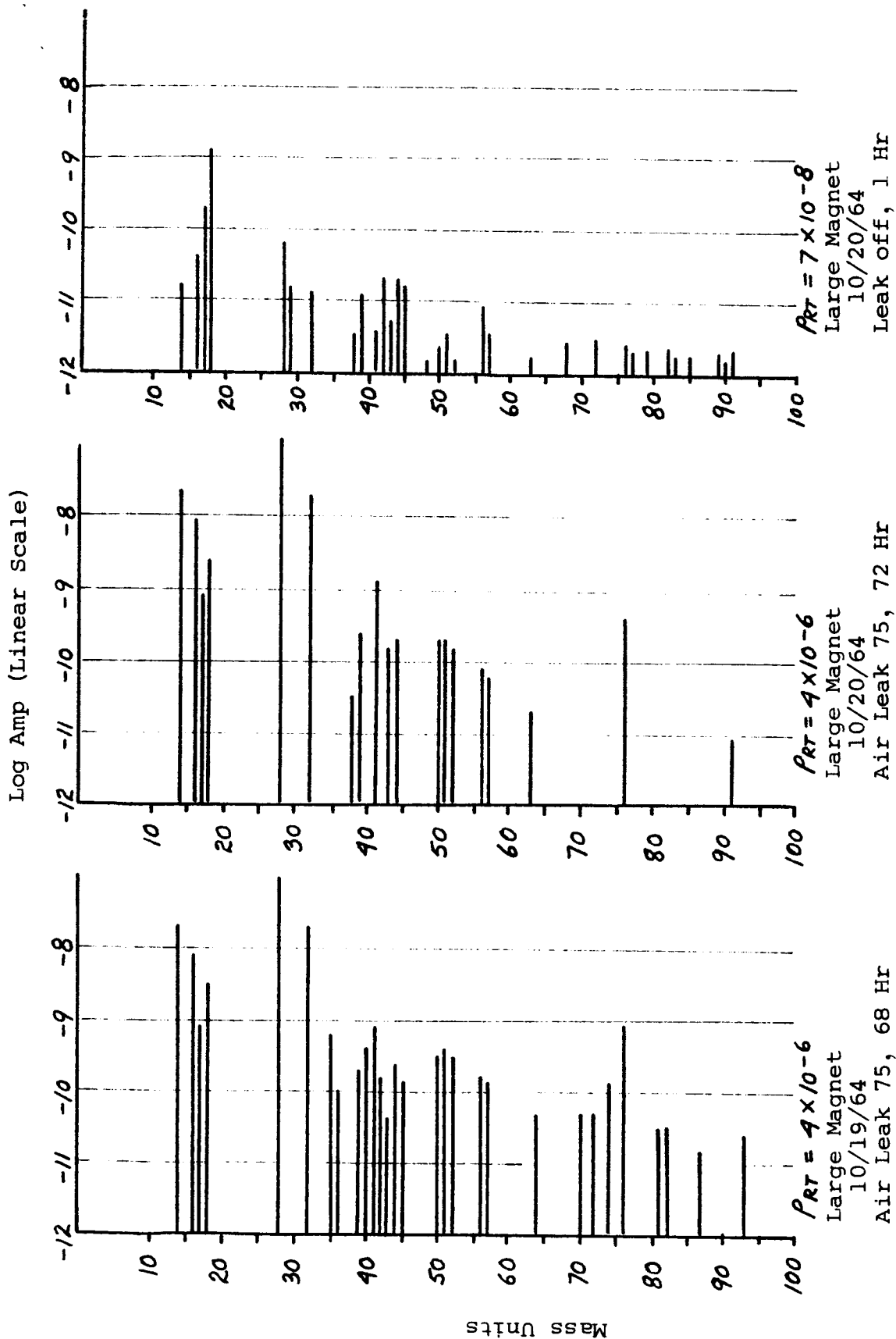
Figure 21

MASS SPECTROMETRY SURVEY FOR STATION 5
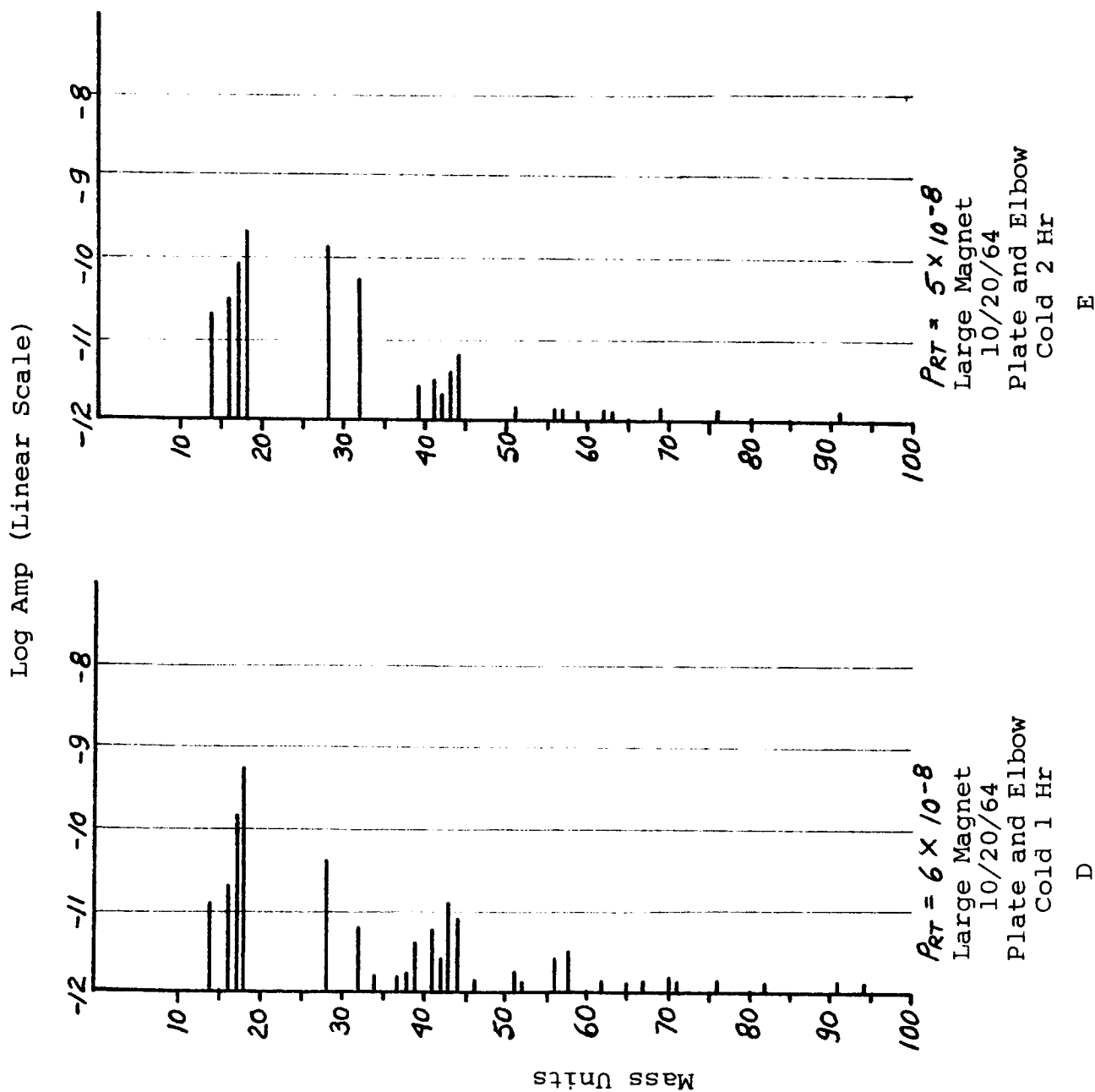
Figure 21 (cont.) MASS SPECTROMETRY SURVEY FOR STATION 5
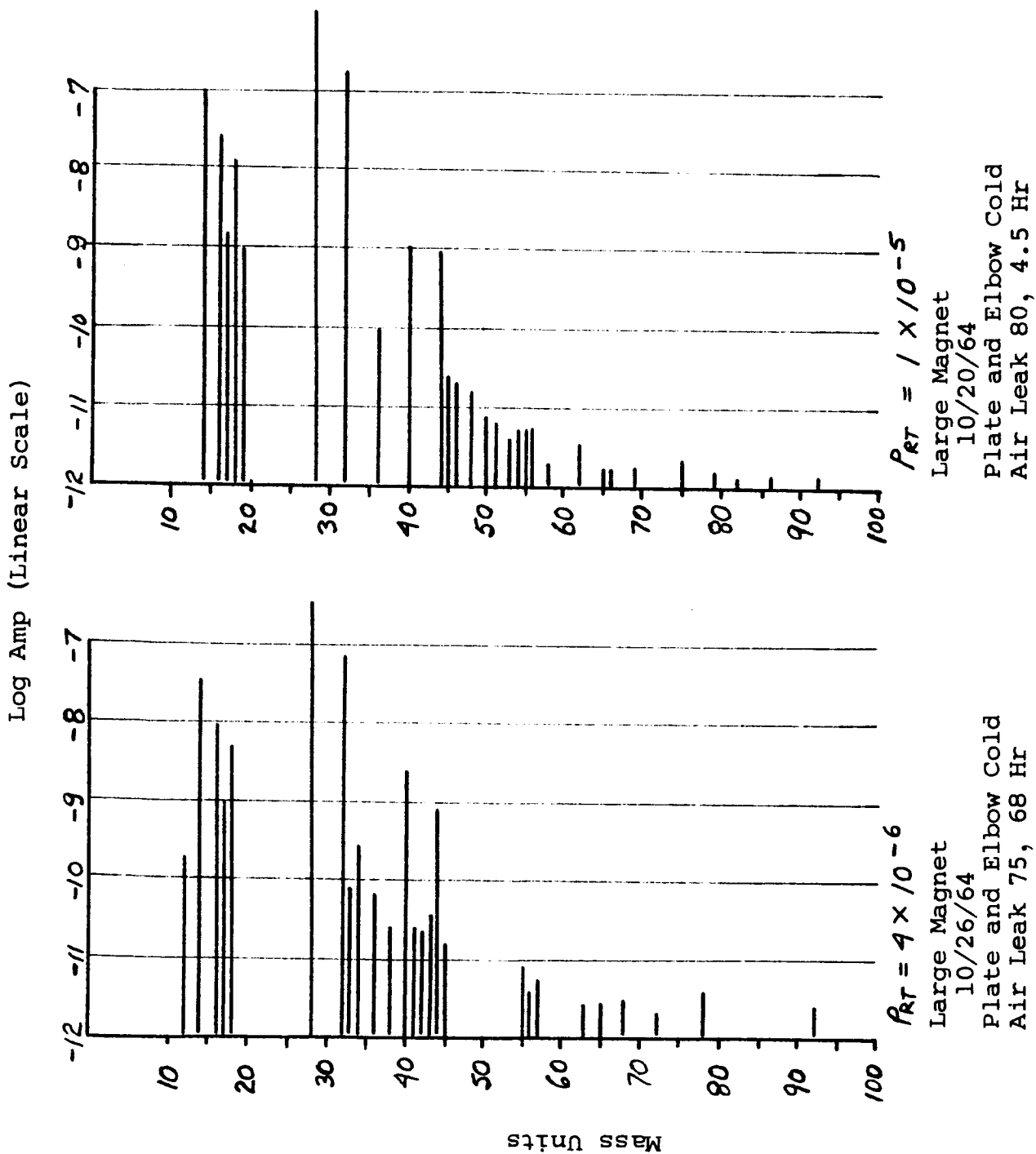
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Log Amp (Linear Scale)

$P_{RT} = 1 \times 10^{-5}$

Large Magnet
10/20/64
Plate and Elbow Cold
Air Leak 80, 4.5 Hr

$P_{RT} = 4 \times 10^{-6}$

Large Magnet
10/26/64
Plate and Elbow Cold
Air Leak 75, 68 Hr

Mass Units

Figure 21 (cont.)

MASS SPECTROMETRY SURVEY FOR STATION 5

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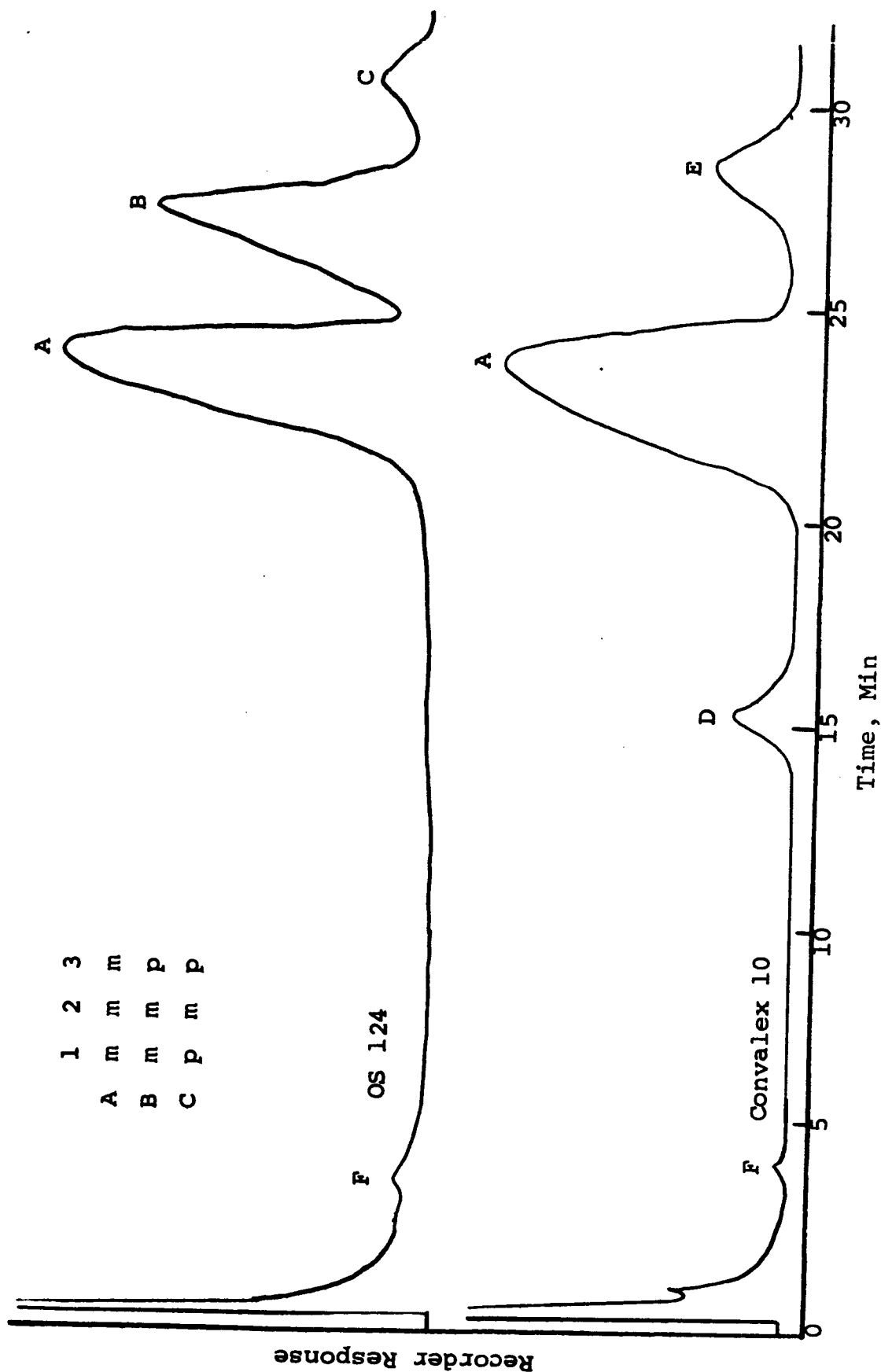
ISOMER DISTRIBUTION OF THE UNUSED POLYPHENYL ETHER FLUIDS
OS 124 AND CONVALEX 10

Figure 22

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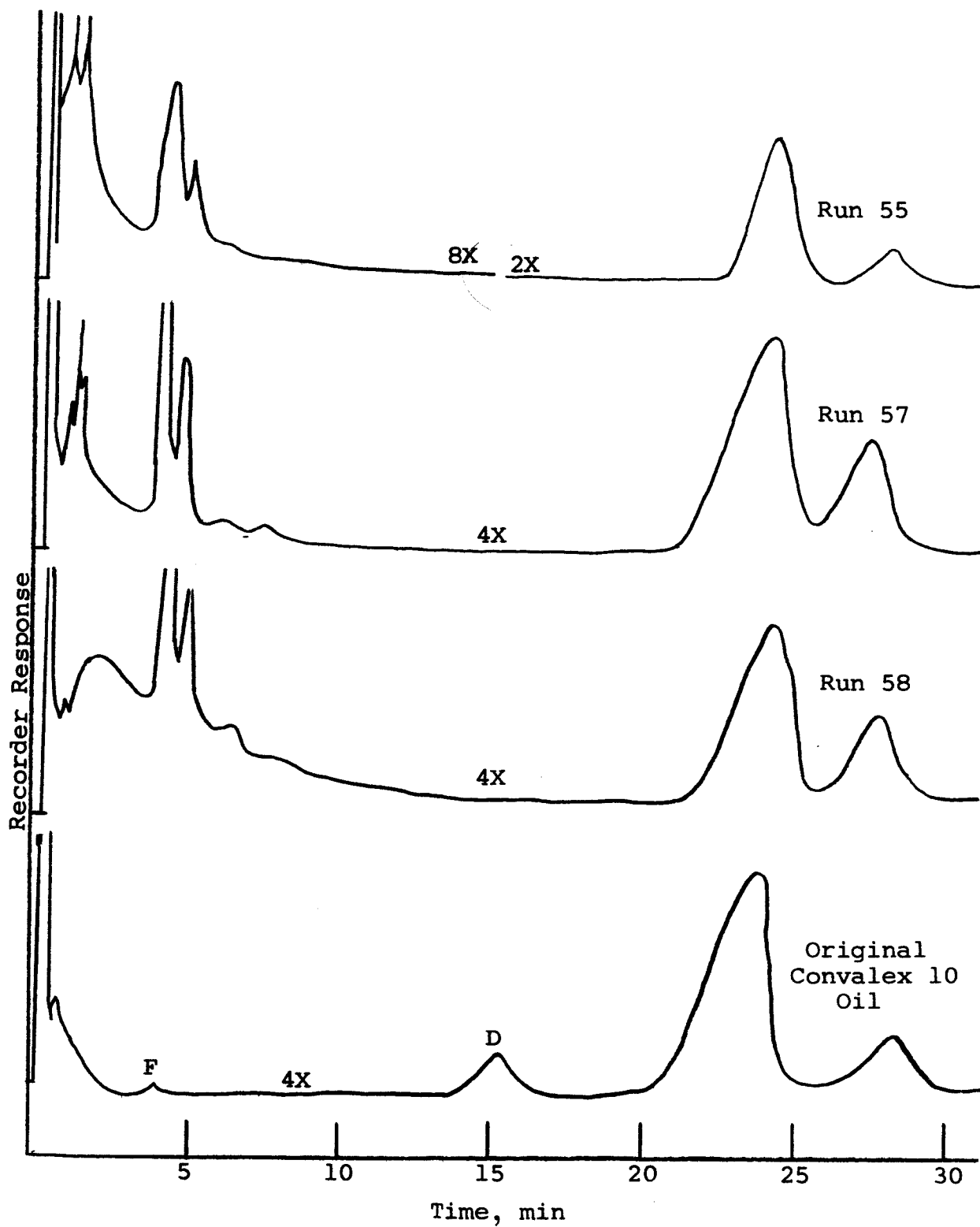
Figure 23

ISOMER DISTRIBUTION IN CONVALEX 10 OIL
USED FOR THREE MONTHS

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ORIGINAL MIXED OIL
1% DC 704

DC 704

DC 705

Loenco Dual Flame
Gas Chromatograph
5% SE 30 Column 6 ft x 3/16 in
Attenuation = 16X
Detention: @ Peak A 3.9 cm
            @ DC 704 7.2 cm
            @ DC 705 17.3 cm

Helium pressure 10 psig
T = 300°C

PEAK
A

SAMPLE 232
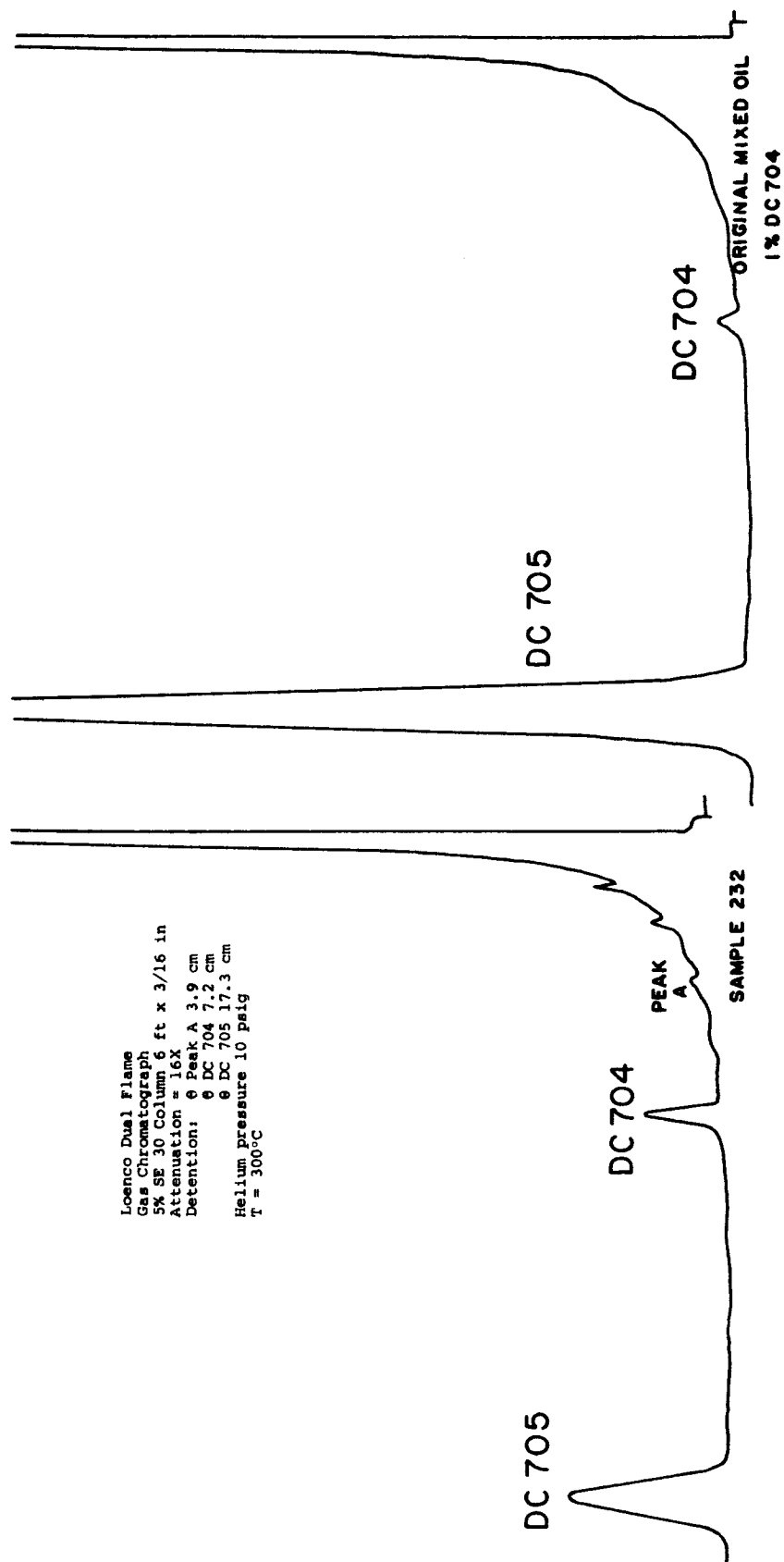
DC 704

DC 705

FIGURE 24   GAS CHROMATOGRAMS FOR MIXED DC 704 / DC 705
BACKSTREAMING SAMPLES

Loenco Dual-Flame
Gas Chromatograph
5% SE 30 Column 6 ft x 3/16 in.
Attenuation = 8X
Detention θ DC 704   5.6 cm
          θ DC 705 13.6 cm
Helium pressure 10 psig
T = 300°C

DC 705

DC 704

Sample 239
Conc.

Figure 24 (Cont.)

GAS CHROMATOGRAPHS FOR MIXED DC 704/DC 705
SAMPLES

156

Loenco Dual Flame
Gas Chromatograph
5% SE 30 Column 6 ft x 3/16 in
Attenuation: 4, 8X
Detention: @ Peak A 5.2 cm
             @ DC 704 7.2 cm
             @ DC 705 17.0 cm

Helium pressure 10 psig
T = 300°C

DC 705

DC 704

PEAK
A

SAMPLE
234 CONC.

DC 705

DC 704

PEAK
A
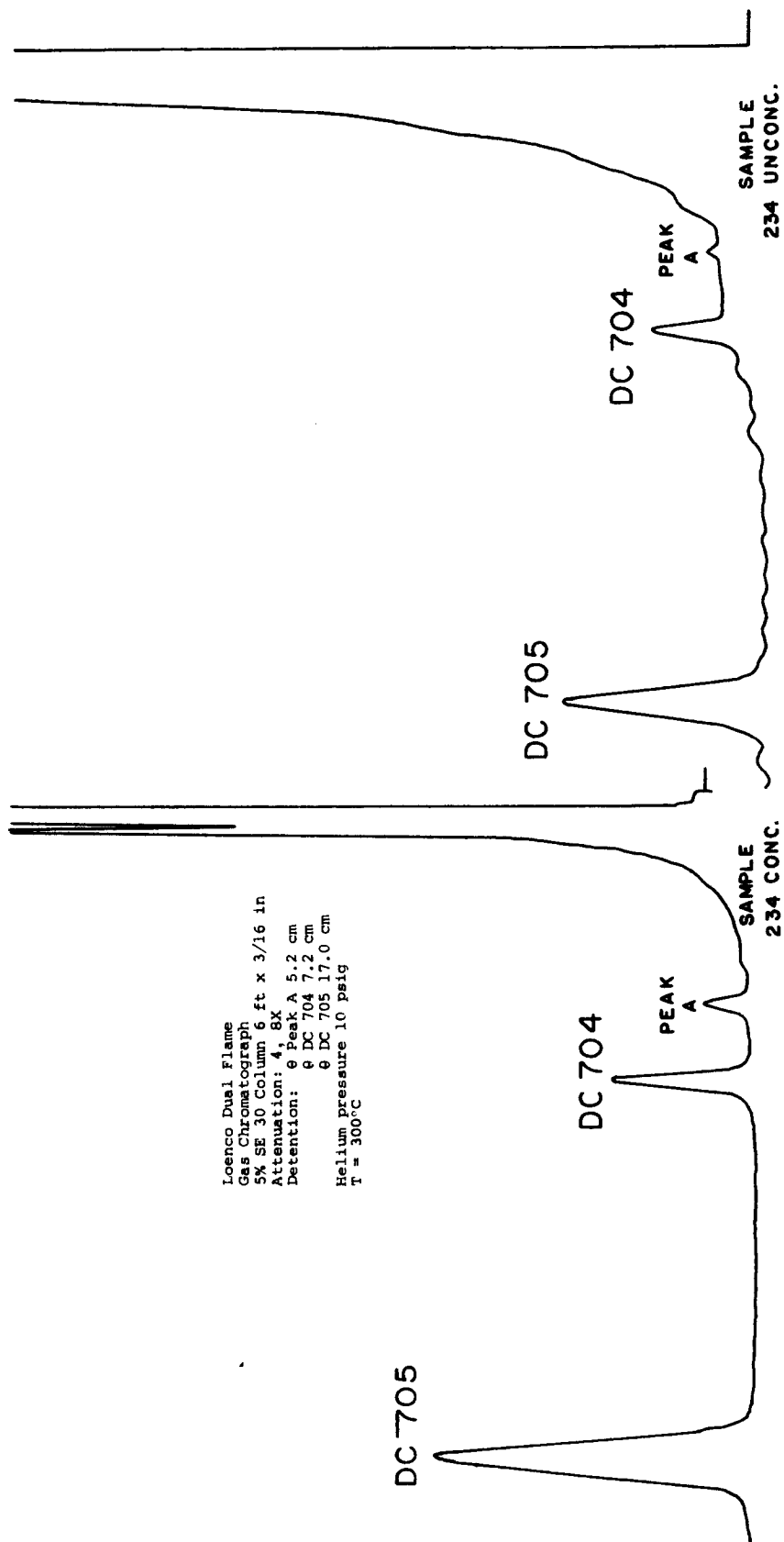
SAMPLE
234 UNCONC.

FIGURE 24 (CONT.)

GAS CHROMATOGRAMS FOR MIXED DC 704 / DC 705
BACKSTREAMING SAMPLES
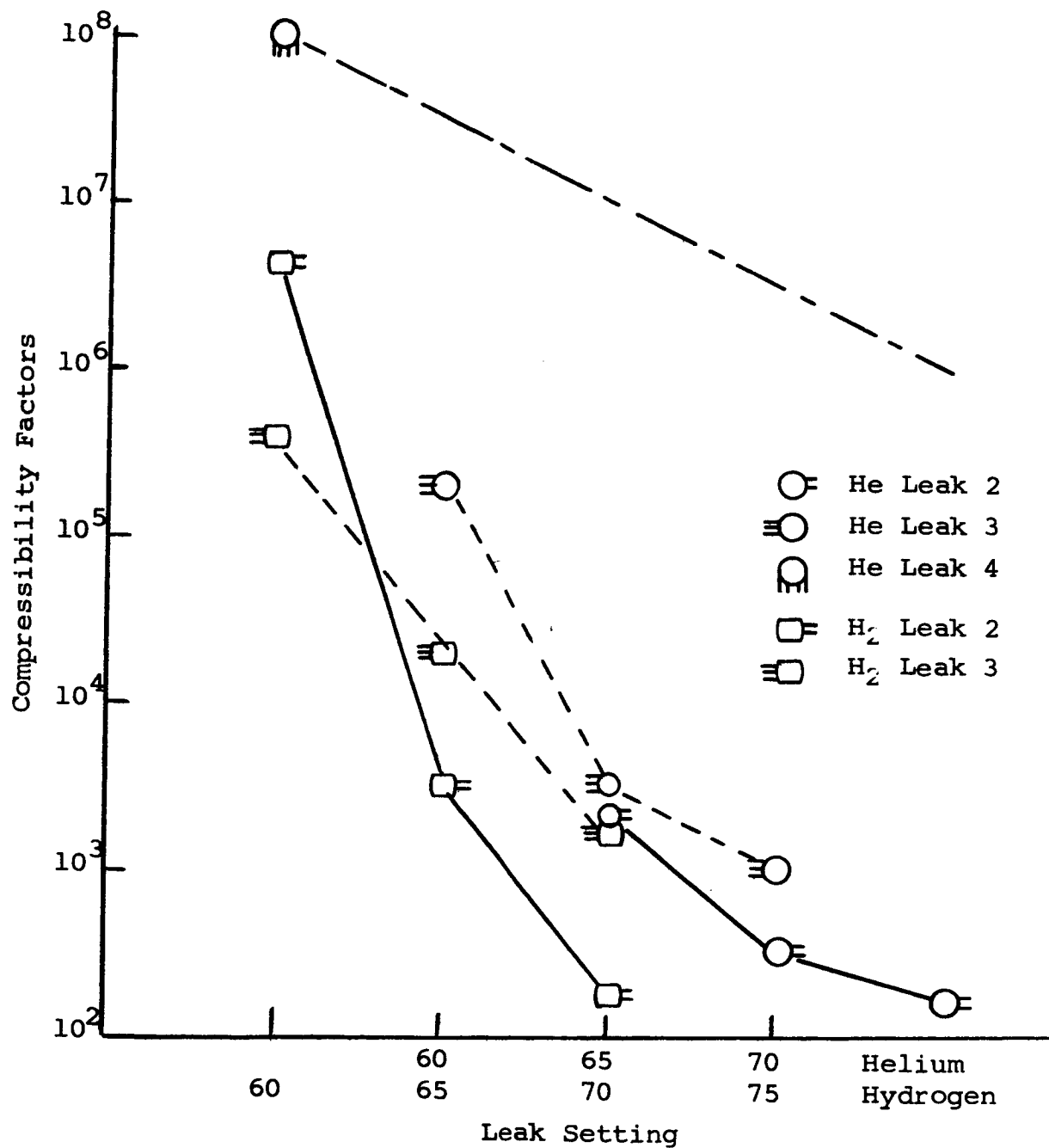
Figure 25

COMPRESSIBILITY FACTORS VERSUS LEAK RATE AND LEAK LOCATION

158
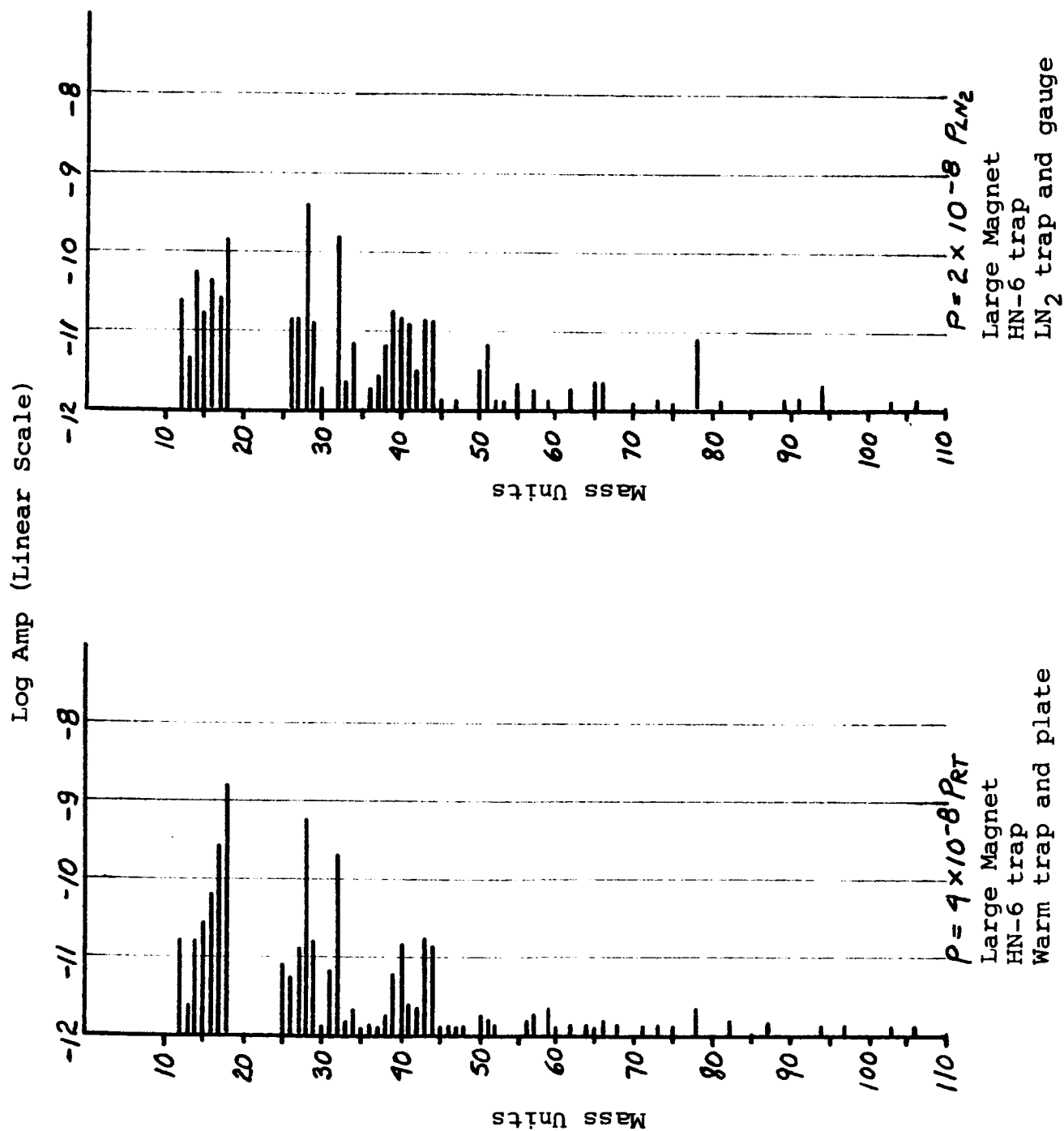
Figure 26

MASS SPECTROMETRY SURVEY FOR STATION 3

159
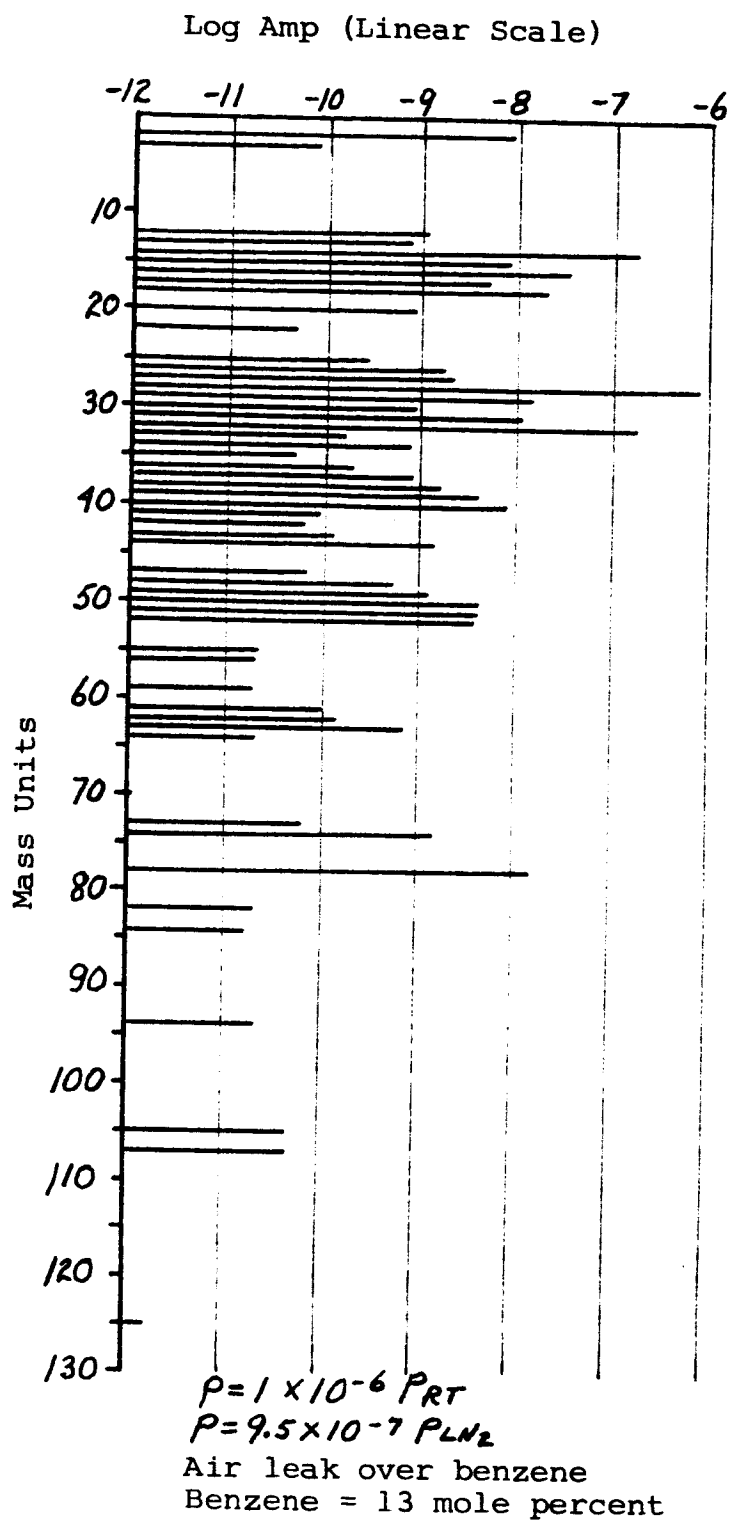
Figure 27

MASS SPECTRUM OF BENZENE

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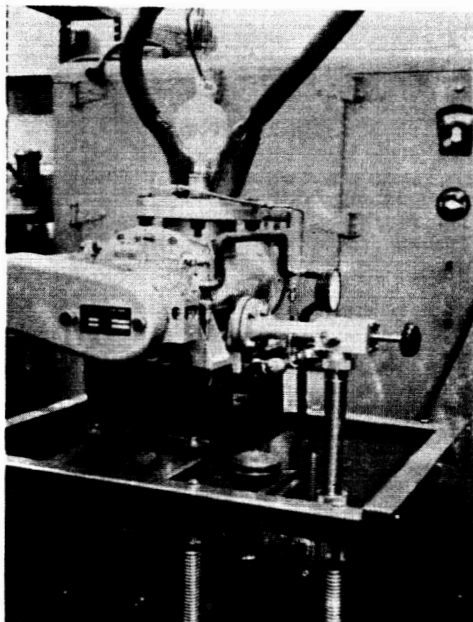
Figure 28

TURBOMOLECULAR PUMP STATION
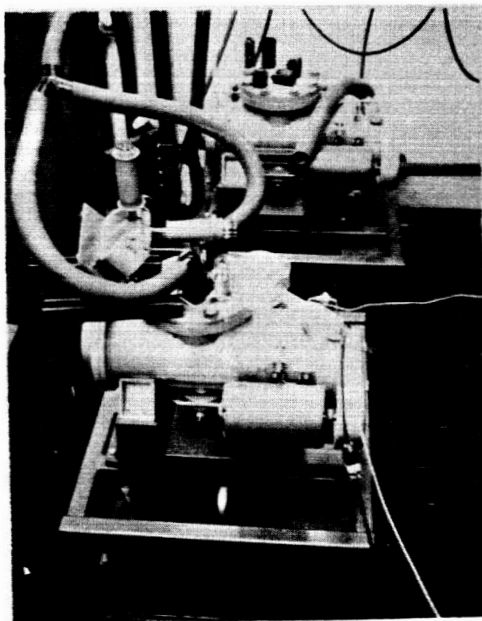
Figure 29

TURBOMOLECULAR PUMP STATION 8

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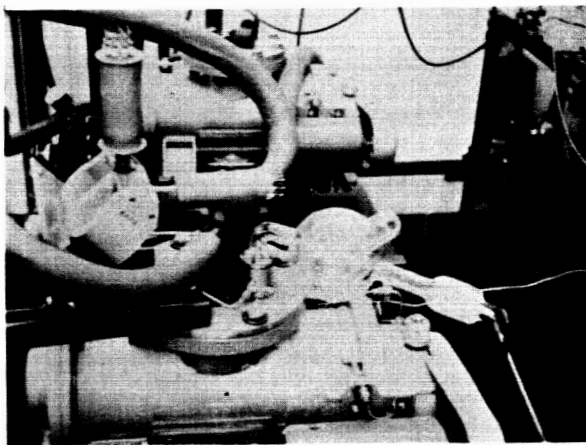
A. Test head attached to
    Station 8
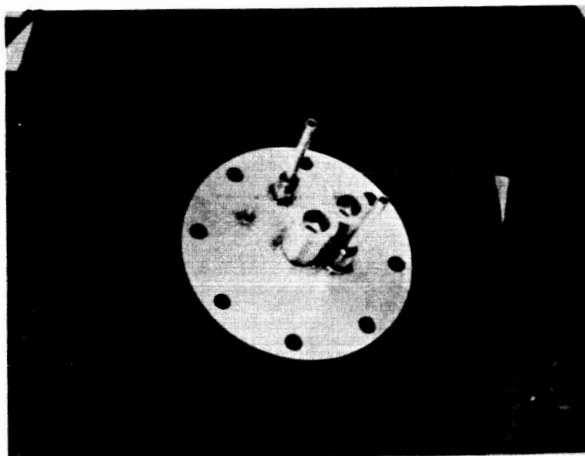
B. Top view of test head.

C. View of head showing
    the collection plate.

Figure 30

TURBOMOLECULAR PUMP TEST HEAD

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RETENTION TIME, CM.
FIGURE 31
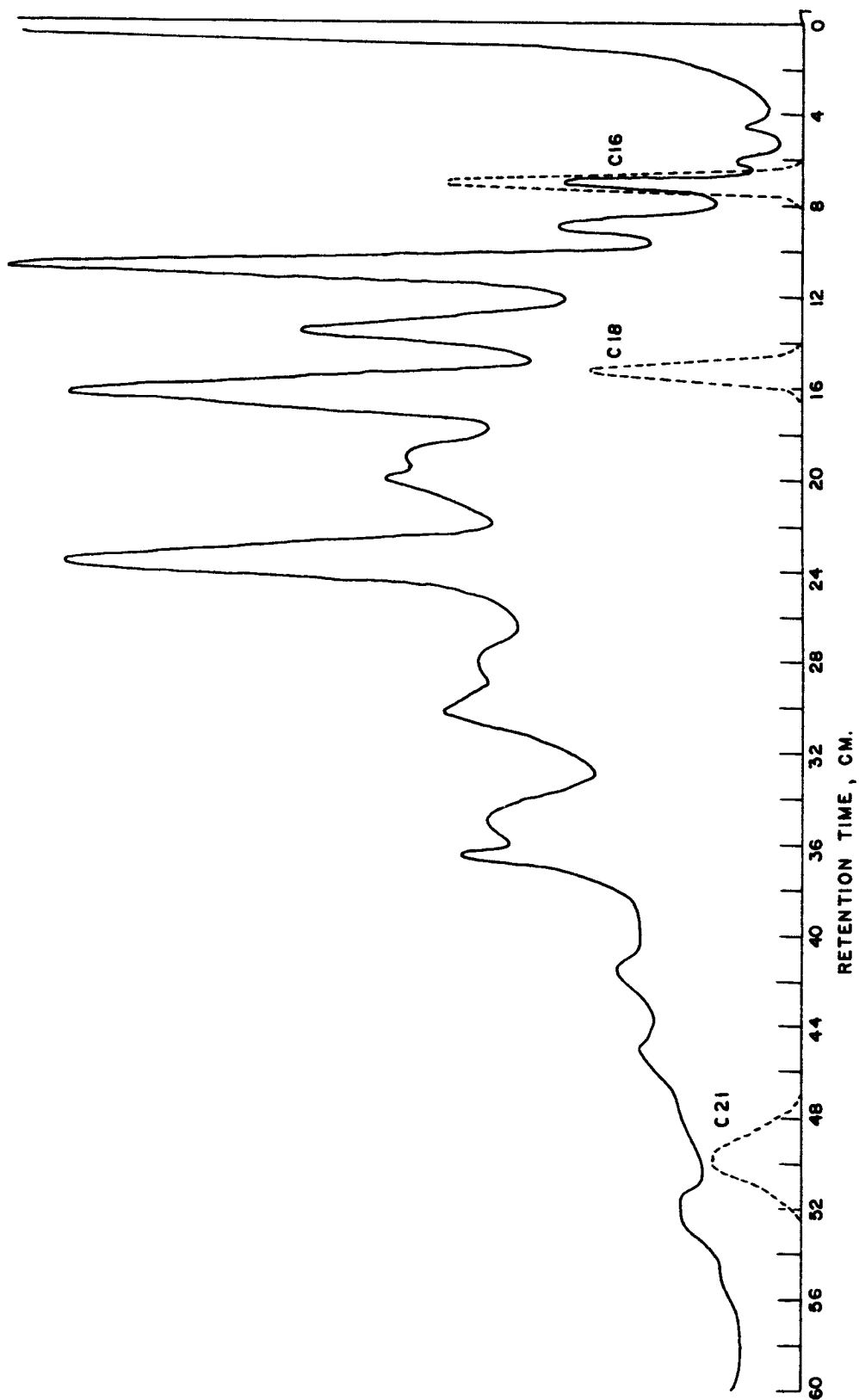
UNUSED TURBO MOLECULAR OIL VERSUS STANDARD STRAIGHT CHAIN
HYDROCARBONS
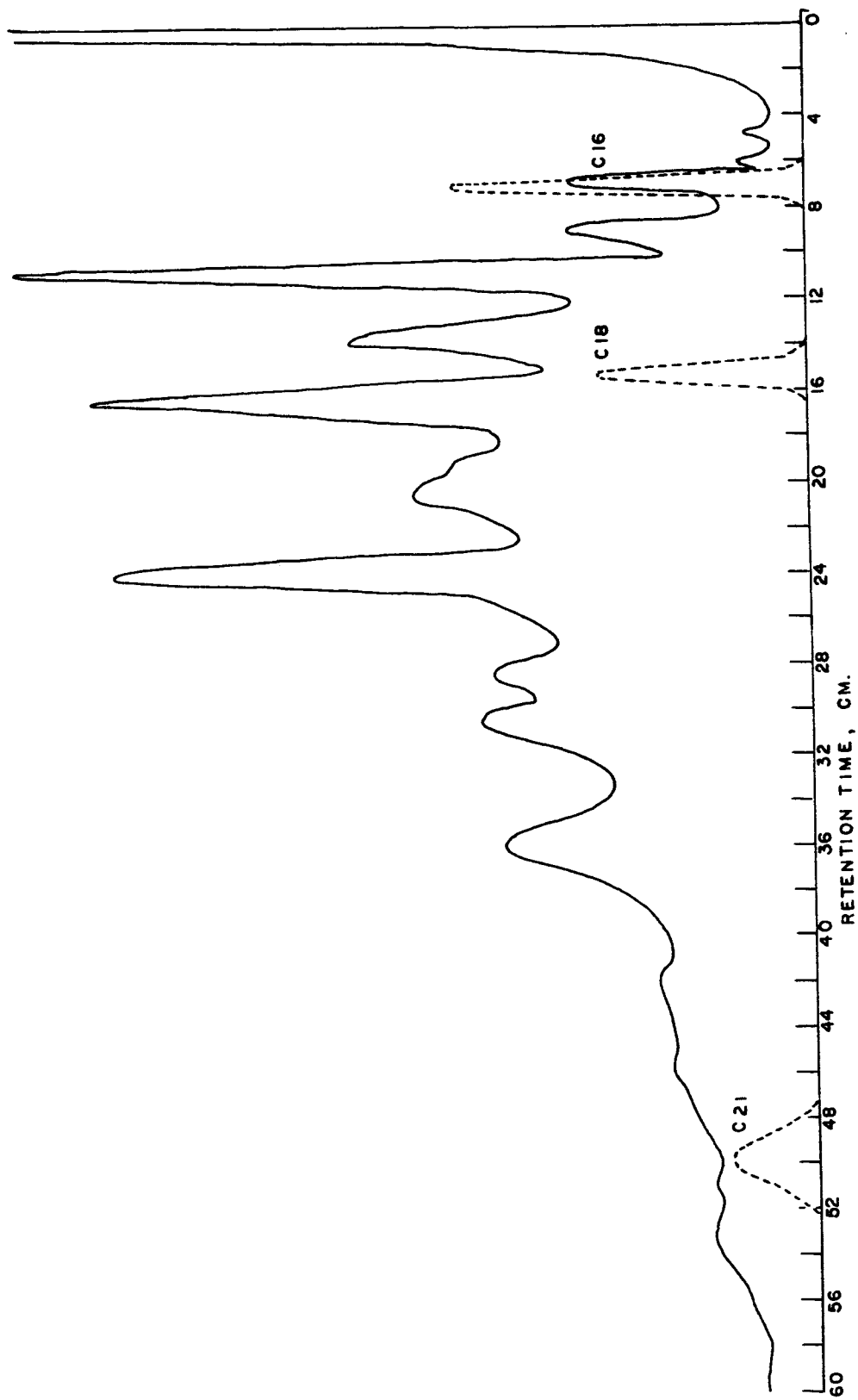
FIGURE 32

USED TURBO MOLECULAR OIL VERSUS STANDARD STRAIGHT CHAIN
HYDROCARBONS

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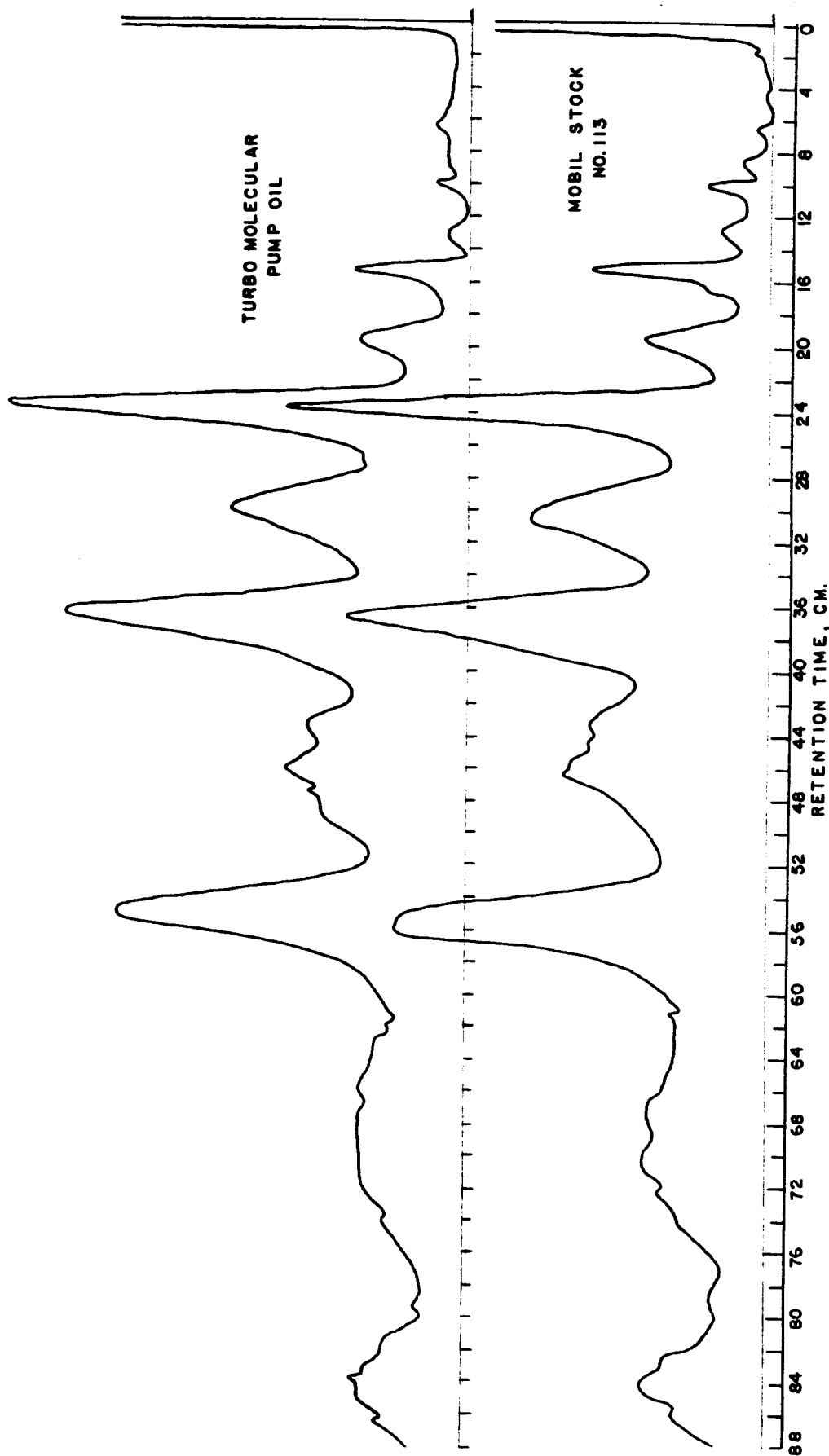
FIGURE 33

COMPARISON OF TURBO MOLECULAR OIL WITH MOBIL 113 STOCK

TURBO MOLECULAR
PUMP OIL

MOBIL STOCK
NO. 113

RETENTION TIME, CM.

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Figure 34

INFRARED SPECTRUM OF TURBOMOLECULAR OIL

Figure 35

INFRARED SPECTRUM OF MOBIL STOCK 113

Wavelength, μ

Transmittance %

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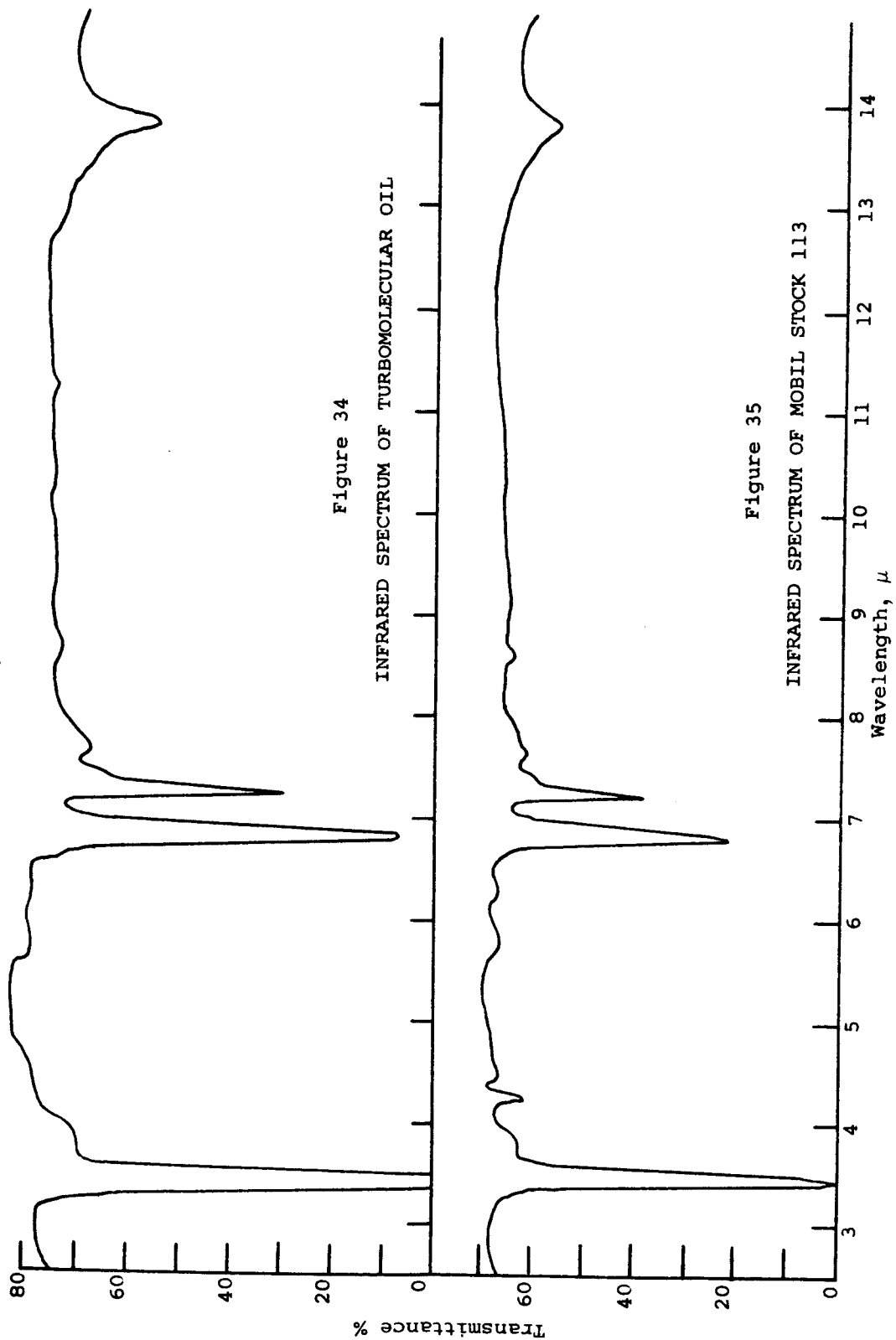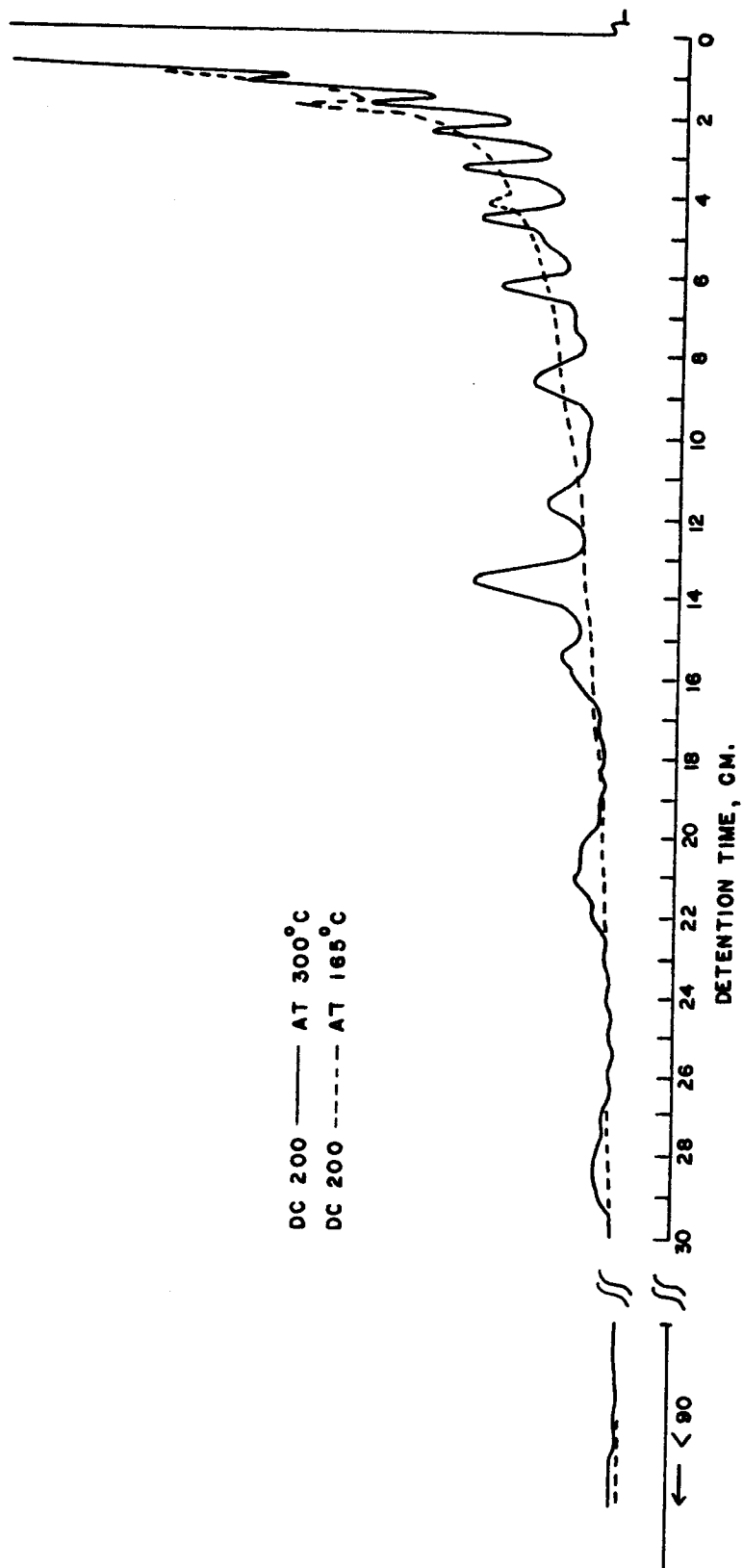
FIGURE 36

GAS CHROMATOGRAMS FOR DC 200

DC 200 ——— AT 300°C

DC 200 ----- AT 165°C

DETENTION TIME, CM.

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STANDARD COMPOSITION

C 16   4.92 μg
C 18   6.96 μg
C 21 11.58 μg

DETENTION TIMES

C15   4.5 cm.
C16   7.1 cm.
C17   11.5 cm.
C18   18.1 cm.
C19   27.8 cm.
C20   43.0 cm.
C21   66.2 cm.

DETENTION TIME, CM.
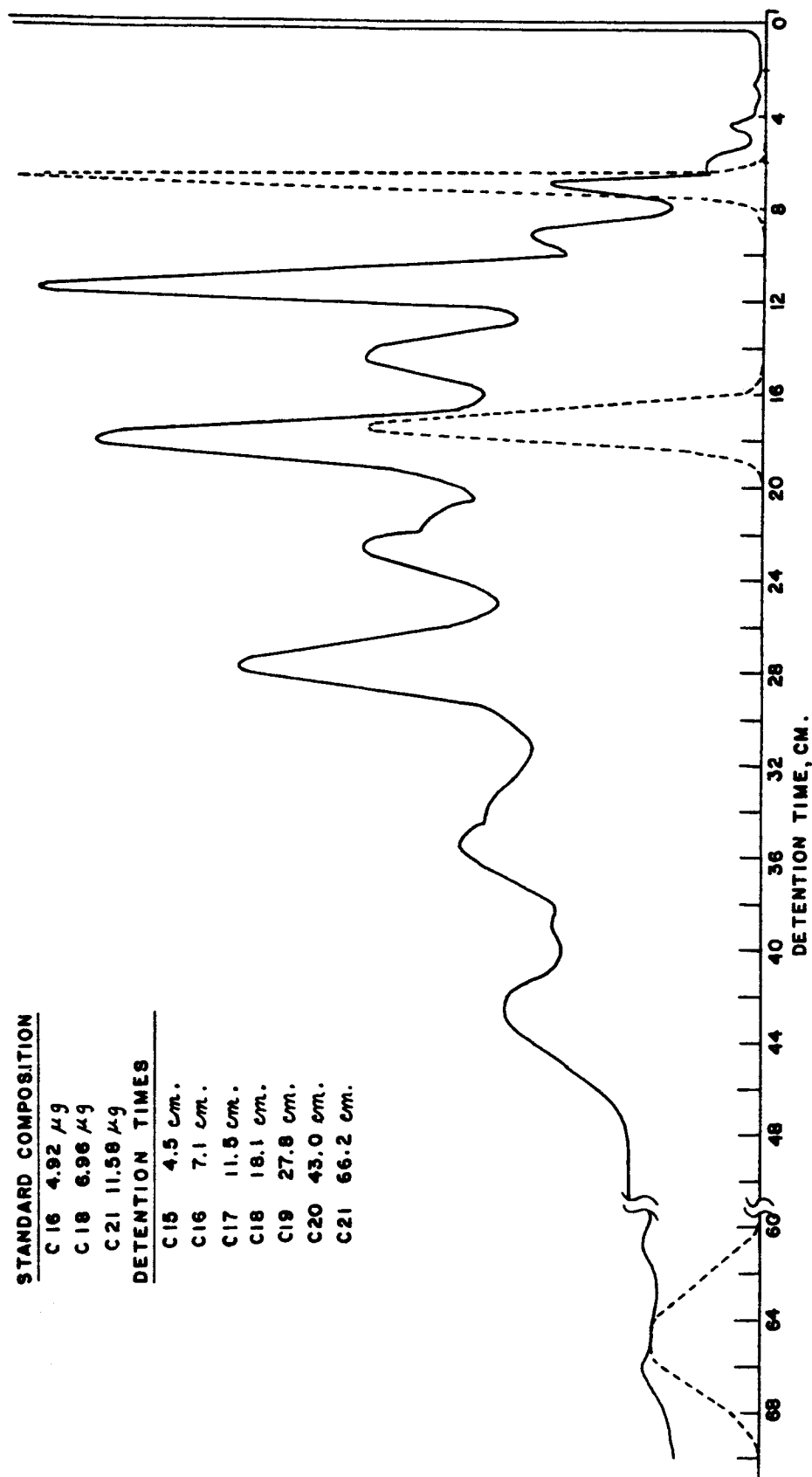
FIGURE 37

GAS CHROMATOGRAM FOR FIRST TURBO MOLECULAR PUMP OIL

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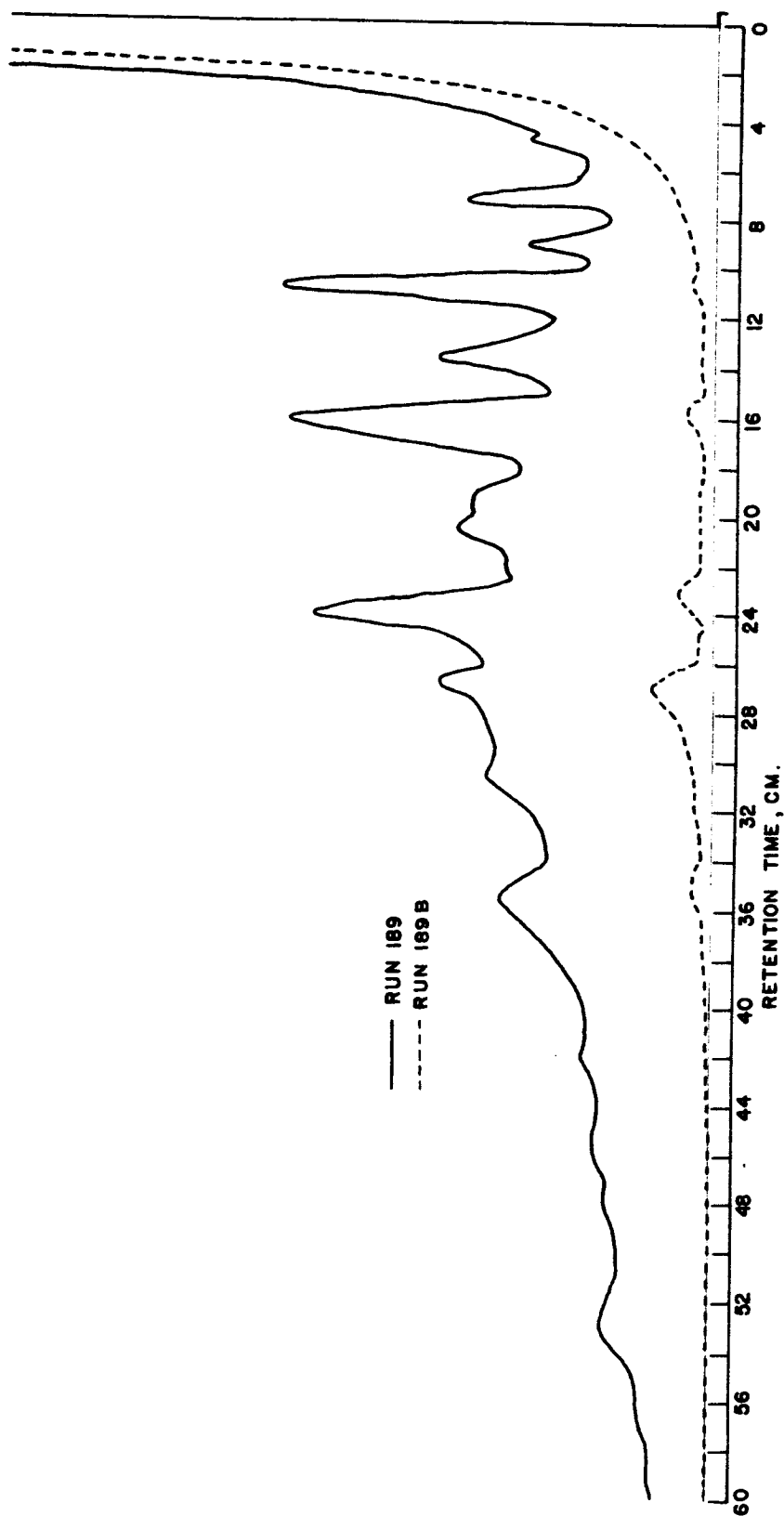
FIGURE 38

GAS CHROMATOGRAMS FOR RUNS 189 AND 189B

——— RUN 189
- - - - RUN 189B

RETENTION TIME, CM.

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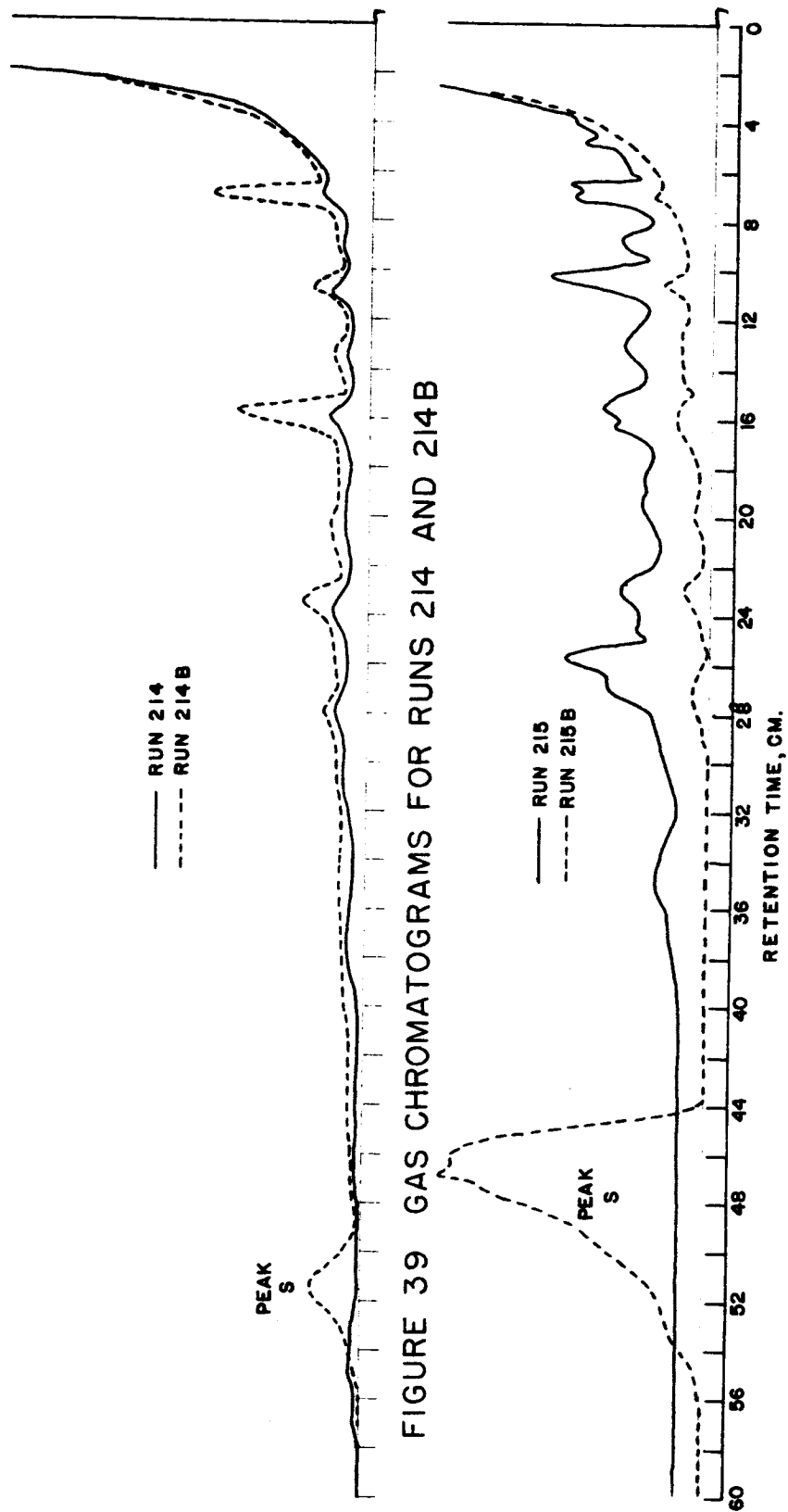
FIGURE 39 GAS CHROMATOGRAMS FOR RUNS 214 AND 214B
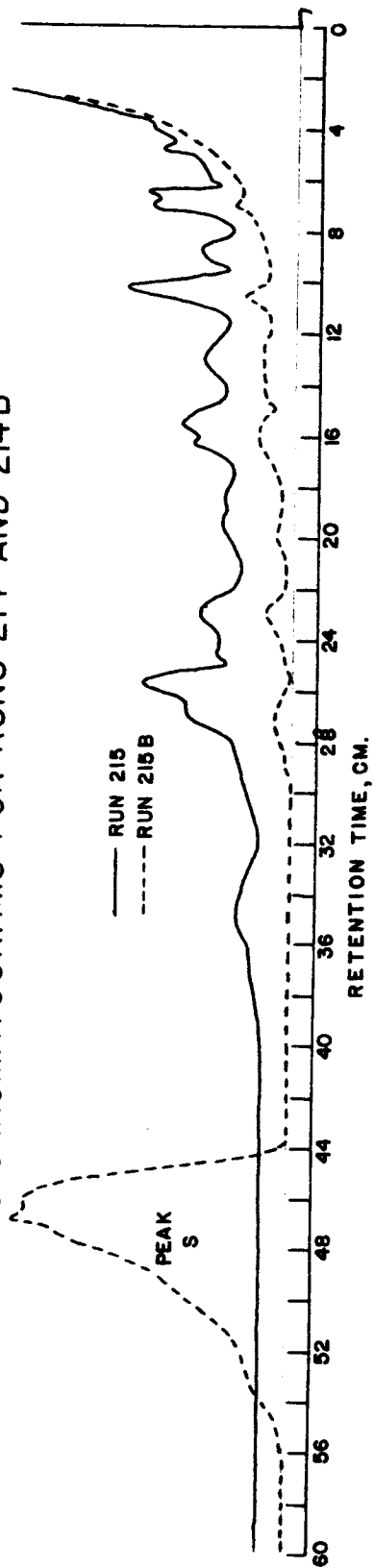
FIGURE 40 GAS CHROMATOGRAMS FOR RUNS 215 AND 215B

170

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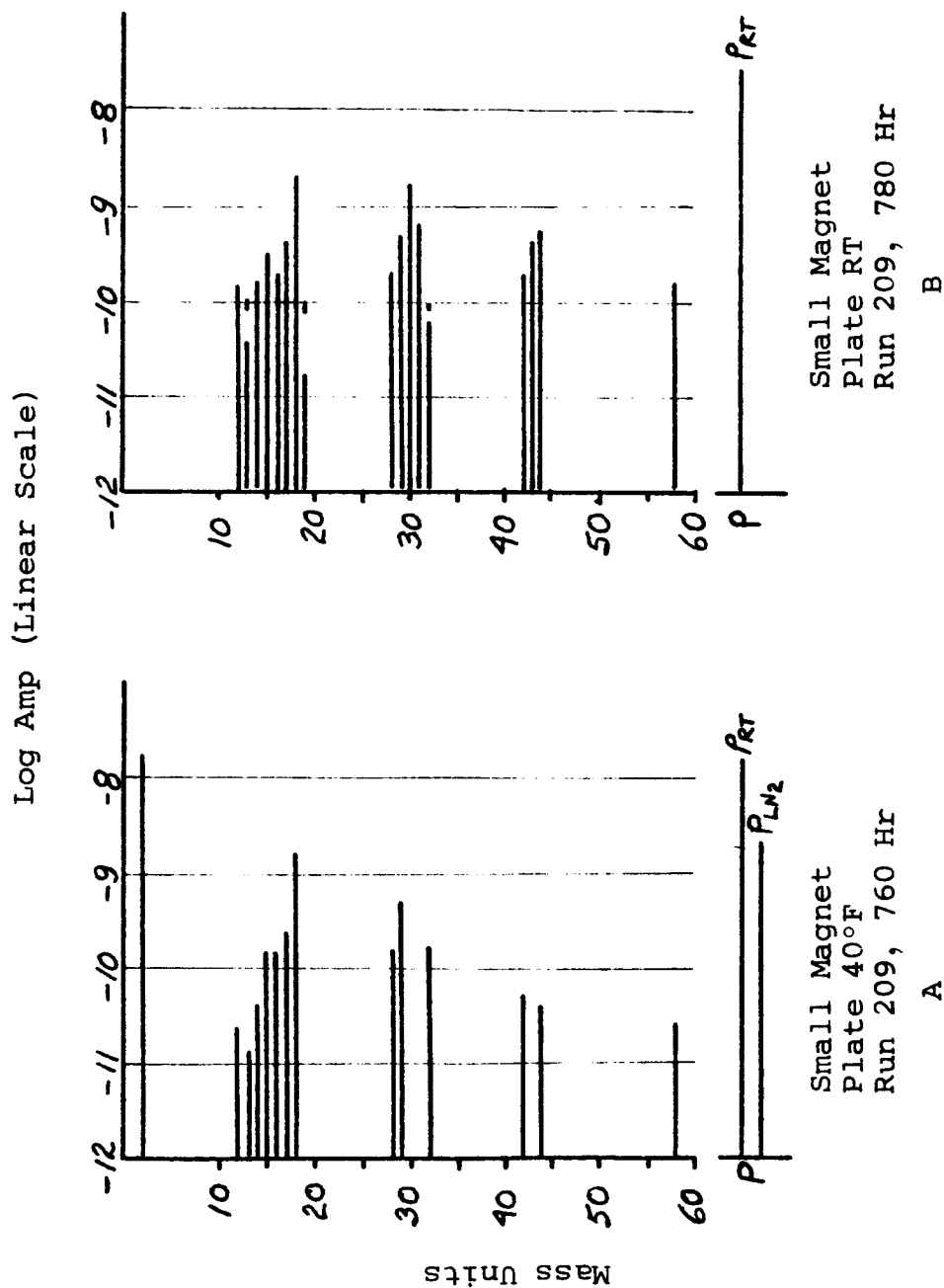
Figure 41   MASS SPECTRA SURVEY OF STATION 8
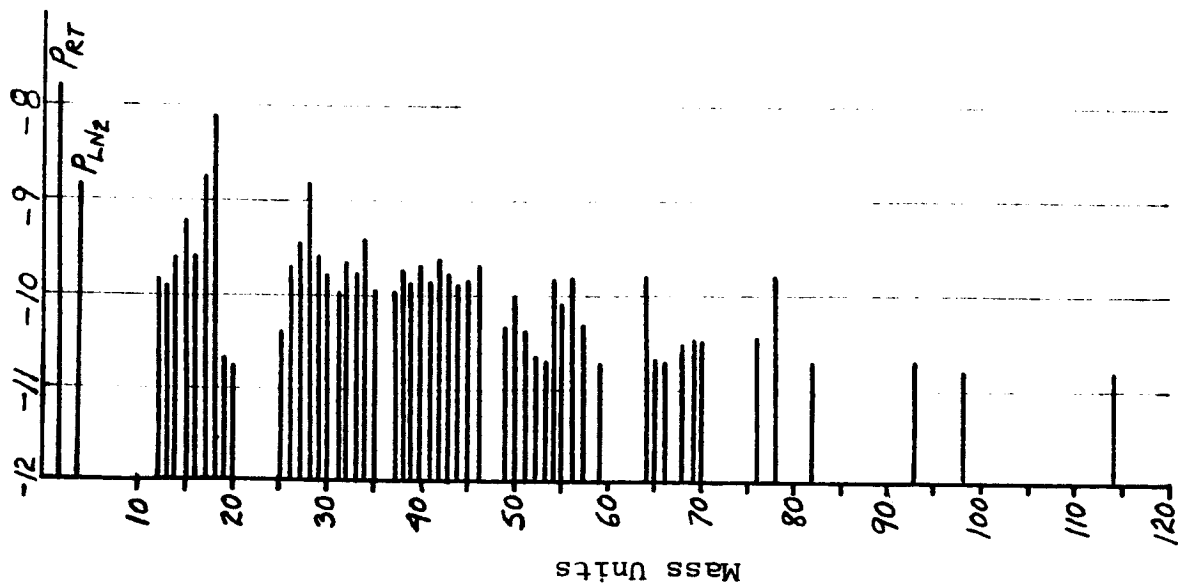
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Figure 41 (cont.)

MASS SPECTRA SURVEY OF STATION 8

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Station 5 OS124 Oil
Plate at -75°F
670 Hr

E

No Trap Large Magnet
Plate at -75°F
Run 220, 500 Hr

D
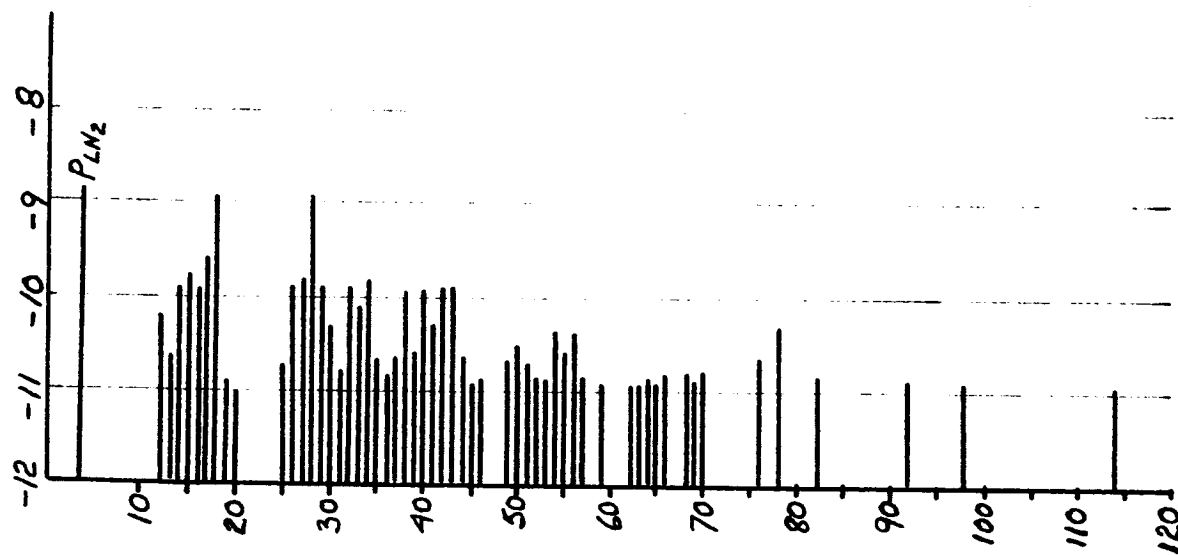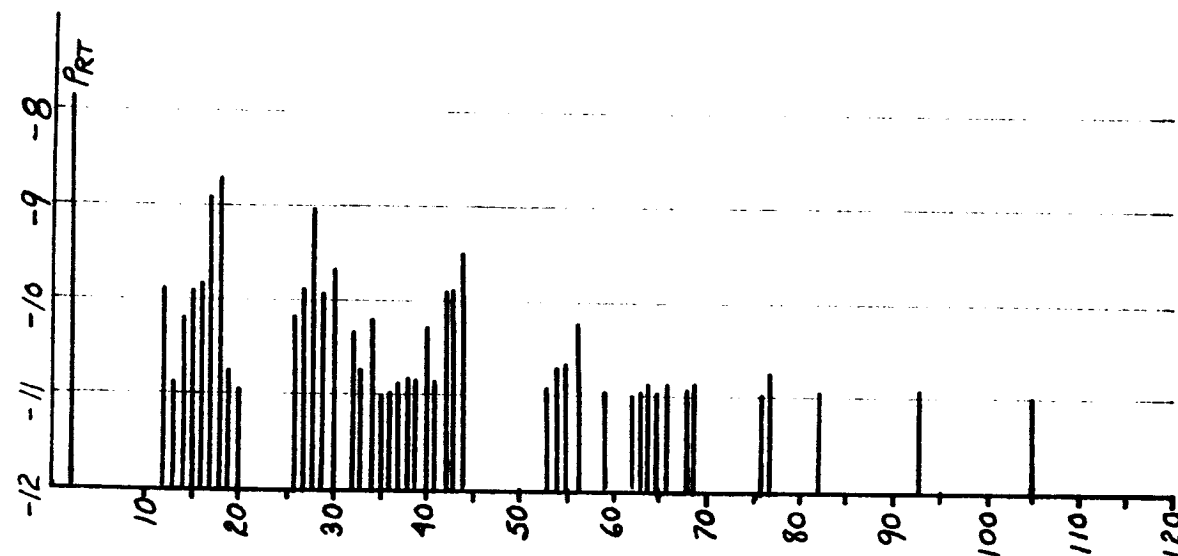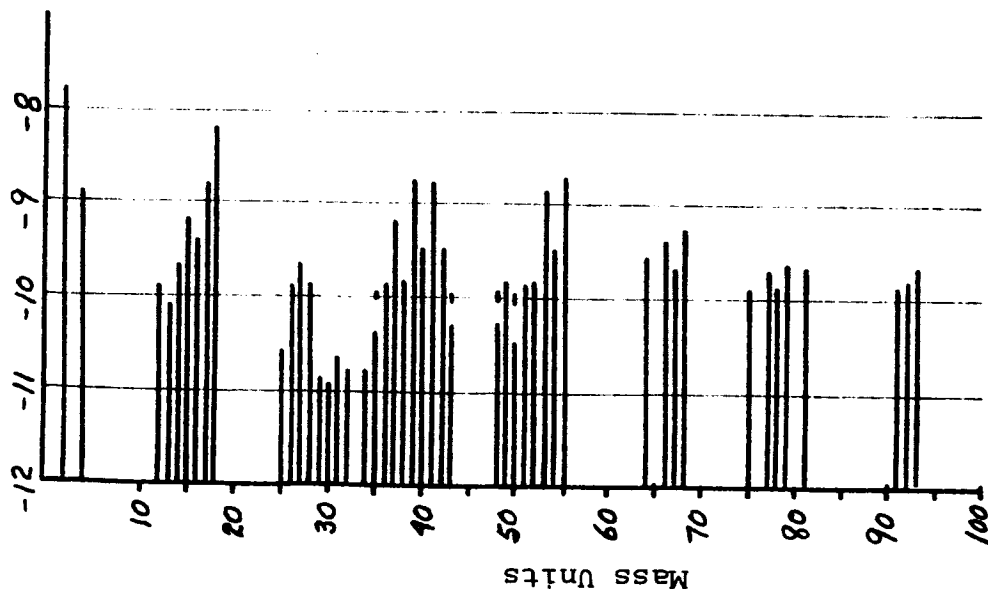
No Trap Large Magnet
Plate at RT
Run 220, 320 Hr

C

Log Amp (Linear Scale)

Mass Units

Figure 41 (cont.)

MASS SPECTRA SURVEY OF STATION 8

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